Metal Progress

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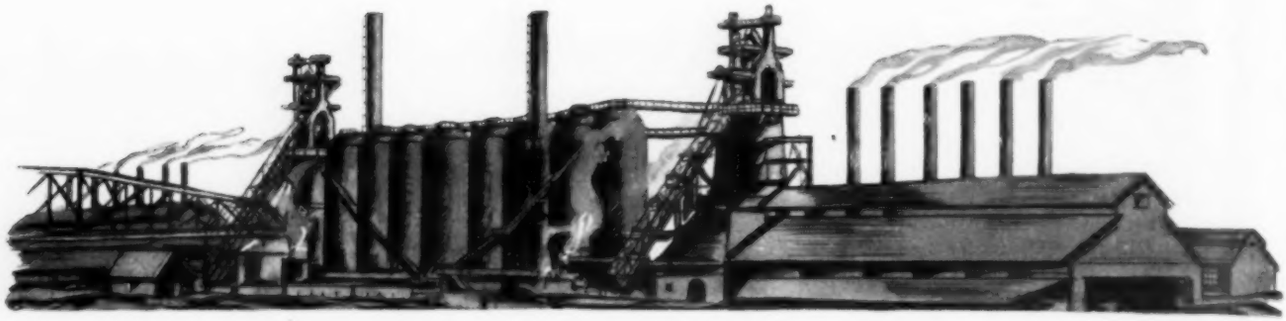

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Commercial Use of Direct Reading Spectrochemical Analysis of Magnesium Alloys

By J. L. Saunderson and T. M. Hess
Spectroscopy Laboratory, The Dow Chemical Co., Midland, Mich.

A PHOTOELECTRIC INSTRUMENT for direct spectrochemical analysis has been in commercial use for about one year in a plant producing magnesium alloys. The apparatus is especially designed to control the composition of the product by analyzing samples from the pots in the shortest possible time. At the present rate of operation, the analysis for seven elements, based upon the average of duplicate determinations, is returned to the alloy floor in less than 5 min. from the time of receipt of the sample. The operation of the instrument is completely automatic from the time the sample electrodes are loaded into the clamps until a record is obtained 40 sec. later; from this record the analysis for seven elements can be read with the aid of calibrated scales. The apparatus is operated entirely with nontechnical personnel, and the analytical results are equal and sometimes superior to those obtained with conventional methods of measurement from a spectrum's photograph. This instrument has been named the "direct reading spectrometer".

In the direct reading spectrometer, special photoelectric tubes replace the conventional photographic plate or film as receivers of light (the spectral lines). An electrical method of computation, which is carried out automatically, is analogous in function to the customary steps consisting of the photographic development, measurement of photographic densities with a microphotometer, and the final graphical calculation of results based upon intensity ratios. The new direct reading method is thus much faster, is less susceptible to human error, and requires fewer and less skilled operators.

In this paper a detailed comparison of the direct reading method with the conventional photographic method is made. Factors such as the relative costs of the equipment, costs per analysis, relative accuracy, and operating personnel are considered. The present and future applications of direct reading spectrochemical analysis are discussed with reference to the type of standard equipment which will be available commercially in a year or two.

Principles of the Method

Since a technical paper describing the direct reading spectrometer, written by J. L. Saunderson, V. J. Caldecourt and E. W. Peterson, appeared last year in the *Journal* of the Optical Society of America (V. 35, p. 681), its operation will be only briefly presented. A simplified schematic diagram is shown in Fig. 1. Sample electrodes, cast in the form of 4-mm. diameter rods, are inserted into clamps and sparked in the usual manner. Light from the spark gap enters the spectrometer, falls upon a concave reflection grating, is dispersed into a spectrum, and is focused along a curve containing several exit slits. Certain selected spectral lines pass through these accurately positioned exit slits and fall upon special photoelectric tubes, known as electron multiplier phototubes, so constructed that the original photocurrent is amplified about 200,000 times within the tube itself. The very weak intensities of light from certain spectral lines thereupon generate currents which are large enough to be handled without complicated external amplification systems required with ordinary phototubes.

During the sparking period, with the switches S closed in the vertical position (see the wiring diagram in Fig. 1), the photocurrents from the multiplier tubes are stored in the condensers C_0, C_1, and C_2. At the end of the sparking period these condensers are charged to voltages which are proportional to the integrated or average intensity of the light of the spectral lines. These voltages must now be measured to establish a correlation with the concentrations of the elements in the sample under test, and this is done in an

indirect manner with the aid of d.c. amplifiers, schematically indicated by triodes in Fig. 1.

At the end of the sparking period, the switches S are automatically thrown to the horizontal position, thus connecting the charge-storing condensers C_0, C_1, C_2 to the discharge resistors R_0, R_1, R_2. The voltage on each condenser then decreases according to the exponential equation

$$V' = Ve^{\frac{-t}{RC}},$$

where V' is the voltage at the time t, V is the original voltage (when $t=0$), e is the base of the natural logarithms, R is the resistance through which the condenser discharges, and C is the capacity of the condenser. Amplifiers are connected to the condensers and are set to operate the relays S_0, S_1, S_2 when the voltages on the condensers C_0, C_1, C_2 fall to some small voltage v. The amplifiers thus perform the function of voltmeters. Because of the high input resistance of the amplifiers, the measurements can be made with a negligible influence upon the voltages being measured.

The "internal standard method" has been found to be the most reliable procedure yet devised in photographic spectrochemical work. This method reduces the effect of fluctuations in the total intensity of the light emitted from the spark by comparing the intensities of the spectral lines of all the unknown elements to the intensity of a spectral line of an internal standard element — most commonly the major constituent of the sample under test, as for example, magnesium in magnesium alloys. Intensity *ratios*, rather than absolute intensities, are used for calibration with composition.

In the direct reading spectrometer the internal standard principle which allows the use of intensity ratios as a basis for the calibration is also used. One of the photomultiplier tubes is placed to receive a selected spectral line of the internal standard element, and the apparatus is adjusted

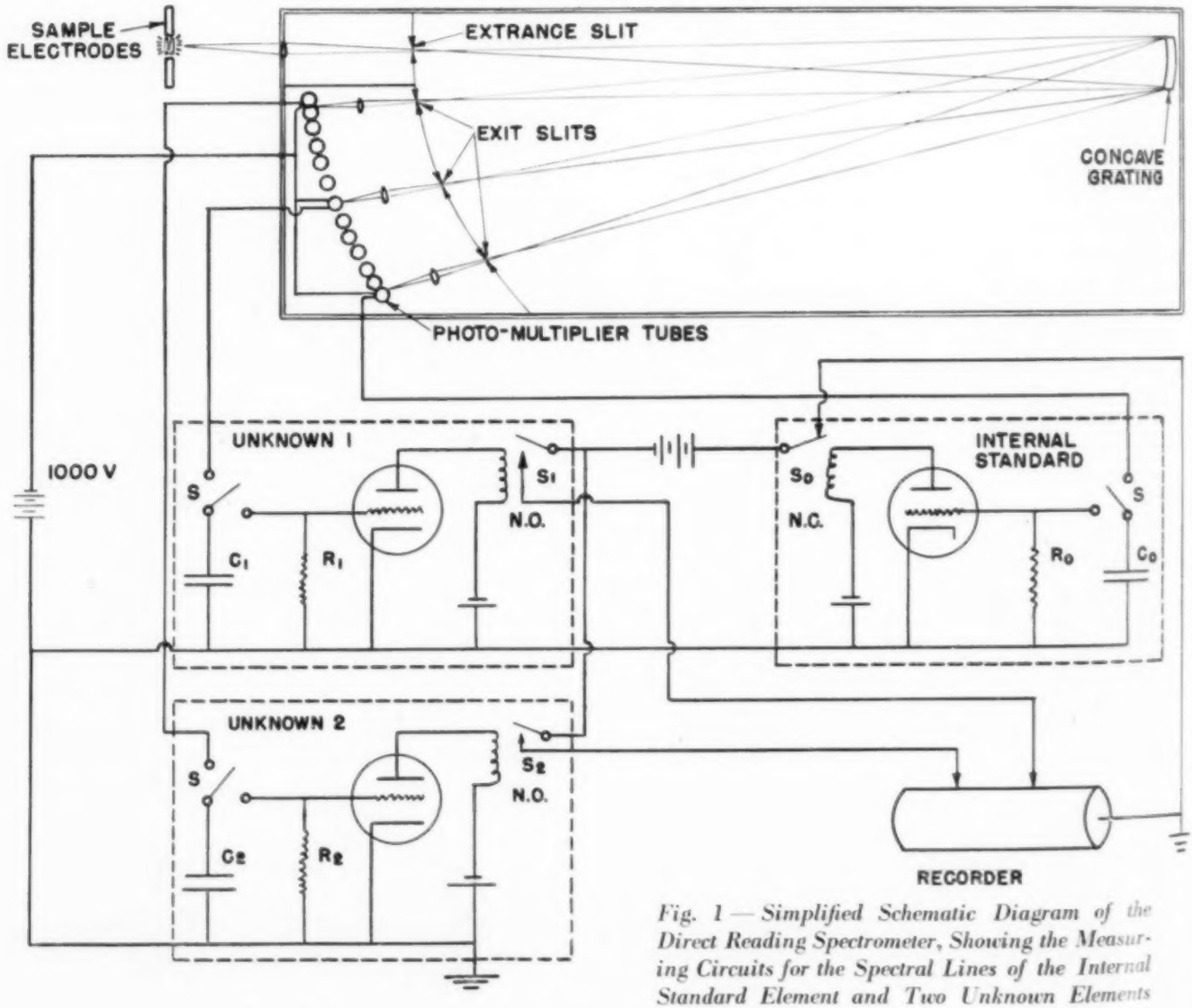


Fig. 1 — Simplified Schematic Diagram of the Direct Reading Spectrometer, Showing the Measuring Circuits for the Spectral Lines of the Internal Standard Element and Two Unknown Elements

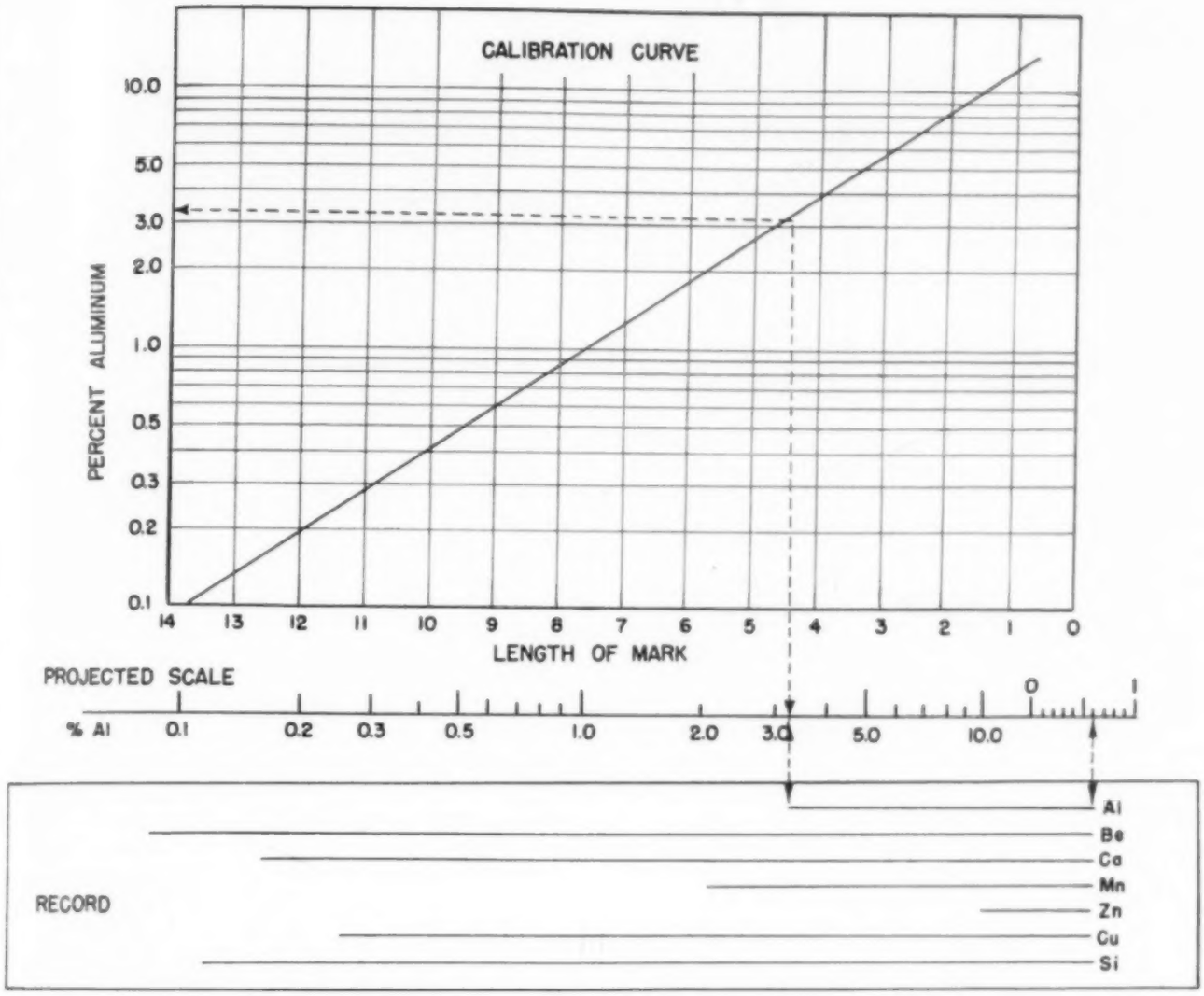


Fig. 2 — Calibration Curve and Projected Scale for Aluminum in a Magnesium Alloy, and a Typical Record Giving the Analysis for Seven Elements

so that the final voltage of the condenser C_0 for the internal standard, after the sparking period, is greater than the voltage on the condensers C_1 and C_2 for the unknown elements. Thus relays S_1 and S_2 operate before S_0, with the result that the two recorder circuits are closed until S_0 operates. Since the recorder carries electrical marking paper moving at constant speed, traces appear on the chart having lengths proportional to the time between the operation of the relays S_1 and S_2 for the unknown elements and the time of operation of relay S_0 for the internal standard element.

The length L_1 of the first mark can be expressed as

$$L_1 = RC \ln \frac{V_0}{V_1},$$

where V_0 is the voltage on C_0 and V_1 that on C_1, and $RC = R_0 C_0 = R_1 C_1$. Since the voltages on the condensers are proportional to the intensities of the spectral lines, L_1 is a measure of the intensity of the spectral line of the first unknown element relative to the intensity of the internal standard line, and the length L_2 of the second mark gives the same relation between the intensity of the spectral line of the second unknown element relative to the internal standard. The lengths of the marks on the record can thus be calibrated in terms of concentrations of the unknown elements.

The direct reading spectrometer in this laboratory has eight amplifiers so that the concentrations of seven elements can be determined simultaneously from the lengths of seven marks on the record. With this instrument additional elements may be determined during a second sparking period, using a new set of multiplier tubes and the same amplifiers and recording system.

A typical record containing seven marks for seven different elements is shown at the bottom of Fig. 2, below a calibration curve and projected scale for aluminum in a magnesium alloy. The calibration curve is established experimentally by plotting the length of the mark obtained with a

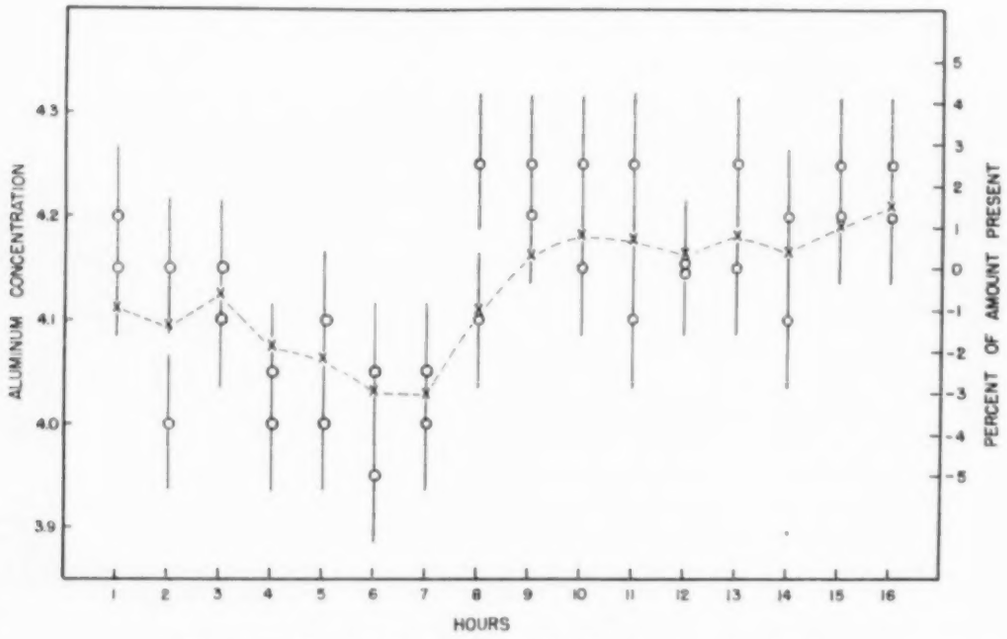


Fig. 3 — Hourly Variation in the Standardization of the Direct Reading Spectrometer Over a Typical 16-Hr. Period

number of samples containing various known amounts of aluminum. When the diagonal calibration curve is projected down on the calibration scale, the latter may be used to read concentrations directly from the record.

Because the calibration for each element is based upon the logarithm of relative intensities, any change occurring in the apparatus causes the inclined line calibration curve to shift parallel to itself without changing the slope. The slope of the calibration curve is determined by the *RC* constant (product of the resistance through which the condenser discharges and the capacity of the condenser) and the characteristics of the spark discharge. Since the slope does not vary, the *calibration* of the projected scale remains valid indefinitely, but it must be located periodically in respect to its *position*.

This periodic standardization is performed by analyzing a standard sample of known composition and measuring the length of the recorded mark with the calibration scale. In standardizing the apparatus, one end of the mark is lined up with the proper concentration figure on the calibration scale and the position of the other end of the mark is read on the small linear "standardization scale" at the right end of the projected scale. This figure is then used for the analysis of unknown samples until the standardization is repeated.

Analysis (Fig. 4) is accomplished by reversing the standardization method — that is, one end of the mark is lined up with the standardization figure (0.6 at the right in Fig. 2) and the concentration read from the calibration scale opposite the other end of the mark (3.3% in the same figure).

This laboratory is at present standardizing the equipment every hour by making duplicate runs on an alloy containing known concentrations of all the elements determined by the instrument. The average of these new duplicate determinations is in turn averaged with the last standardization figure used, so the effects of the random errors are reduced. Data shown in Fig. 3 illustrate the variation in standardization occurring over a typical 16-hr. period; ordinates give actual concentration at left and per cent of the amount present (rather than in the arbitrary figures of the standardization scale) at right. Each individual standardization run is plotted as an open circle, and through each such point is drawn a line of length equal to twice the standard deviation for a single determination. The crosses show the drift in the standardization figure obtained by the averaging method described above. Under the present operating schedule,

Fig. 4 — View Showing the Recorder and Use of the Projected Scales for Reading Percentages From the Record

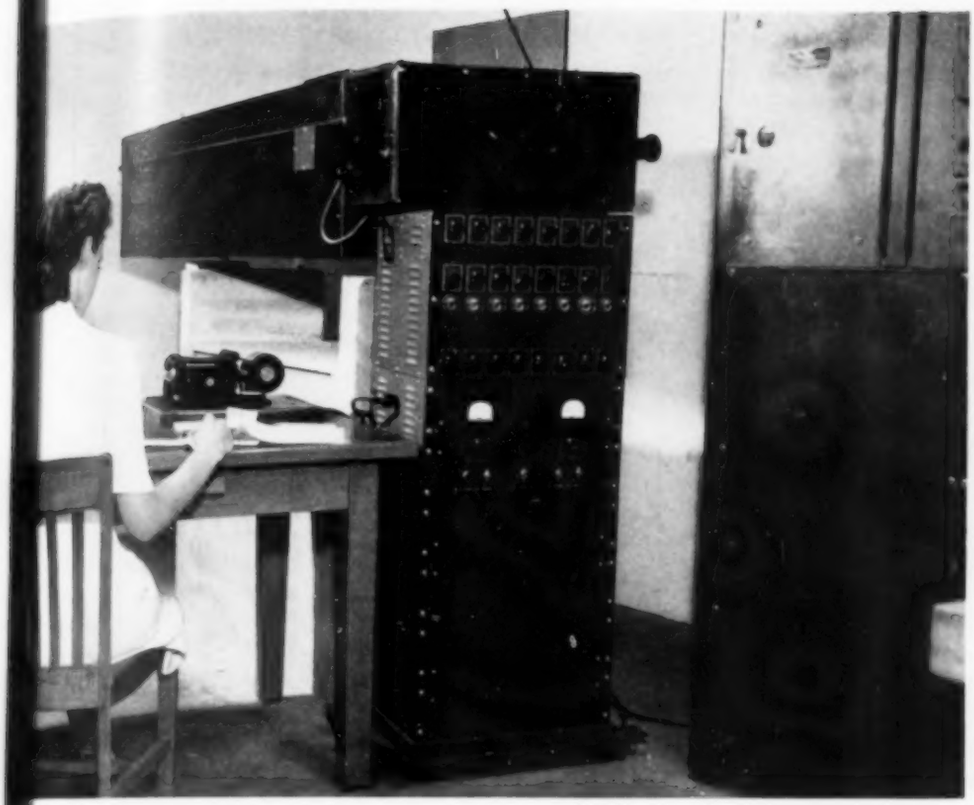


Fig. 5 — View of the Direct Reading Spectrometer. The spark excitation unit is partly shown at the right while the grating housing and phototube compartment appears in the center with the recording chassis below. The recorder is visible on the table in front of the operator

about 5 min. out of every hour is spent in running and averaging the two standardization samples.

Variations in the standardization figure result from slight shifts in the positions of the slots, dust on the optical elements, and variations in the supply voltages to the measuring circuits and spark excitation unit.

Samples are received from the alloying floor through pneumatic tubes and are turned down to a uniform diameter on a simplified lathe. At present the samples as-received are only roughly in the form of rods, so that the machining operation requires about 2 min. This can be appreciably shortened by drawing molten metal into a glass tube or using some other casting technique yielding samples of fairly uniform diameter which would require only polishing off the ends. Inasmuch as so much time is now spent machining the whole sample, both ends of two electrodes are prepared for sparking and each sample is run in duplicate. A 5-sec. pre-spark period is used, with the shutter to the apparatus closed, followed by a 20-sec. sparking period during which the multiplier tubes charge the several condensers. These time intervals are controlled by two timers on the front of the spark excitation unit. (The edge of one of these is shown at the upper right corner of Fig. 5.) The spark "exposure" period is followed by a recording period of about 15 sec. Thus, in 40 sec. after starting the automatic sequence, the operator may remove the record and begin reading off the percentages of the various elements from the calibrated scales.

The usual procedure is to reverse the two electrodes at the completion of the spark period so that the second run may begin immediately after the first recording is completed. The results can be read from the record at the rate of something like 5 sec. per element. With only one operator, the total elapsed time between receipt of the sample and reporting the concentrations of seven elements, based upon duplicate determinations, can be as little as 5 min. If two operators are working, the capacity of the apparatus is approximately doubled but the total elapsed time for each sample is not appreciably affected. Improvements in the sample preparation and in the recording method will probably reduce the elapsed time by a factor of two. The present staff of the laboratory consists of one supervisor in charge of the nontechnical routine operators, and one operator on each shift except during rush hours when a second operator is needed.

Analyses for aluminum near 3% obtained by spectrometer by routine operators on commercial samples are compared with results of quantitative chemical methods in the following frequency data for 243 samples. These data thus include the errors of both methods. In fact, of the

Deviation From Chemical Result	Number of Samples
−0.3	1
−0.2	18
−0.1	46
zero	84
+0.1	66
+0.2	23
+0.3	5
+0.4	0

243 samples tabulated, seven doubtful samples were rechecked chemically; in each the second chemical result differed from the first by amounts varying between 0.1% and 0.6%, and each changed in the direction of better agreement with the spectrochemical result. In view of this situation, the tabulation shows exceptionally good agreement between the two methods, with only 2.5% of the results differing by more than 0.2% (two units in the tenth's place). The superiority of the direct reading method over the photographic method is more precisely demonstrated later.

Direct Reading Versus Photographic Methods

Cost of Equipment — A complete laboratory employing the direct reading method can be equipped for about the same amount of money as a photographic spectrochemical installation. The newer installation is essentially complete with an electrode preparation device, a spark excitation unit, a spectrometer equipped with exit slits and electron multiplier tubes, and a recording mechanism. Equipment for electrode preparation and source excitation serve the same purpose as in the photographic method and thus have no reason to differ from the standard equipment now available. The spectrometer is somewhat more complicated than a spectrograph for photographic work, although for routine use it might well be constructed without any adjustable features.

The most complicated part of the direct reading spectrometer is the recording unit, which measures the intensities of the spectral lines and computes the percentages. It replaces the darkroom, the plate or film processing equipment, the microphotometer or densitometer for measuring the blackness of the developed spectral lines, and the calculating board. These items required for the photographic method should just about balance the cost of a recording unit, so that it is expected the two methods will be quite comparable in first cost.

Air conditioning is desirable for a direct reading spectrochemical laboratory from the standpoint of general cleanliness and for protection from the corrosive vapors commonly present in foundries. In localities subject to high humidity, air conditioning is a necessity to insure reliable operation of the electronic equipment at high voltages. An arrangement of the required equipment of a laboratory utilizing a direct reading spectrometer is shown in Fig. 6.

Accuracy — A strict comparison of accuracy between the direct reading and the photographic methods is difficult to make, for it is complicated by the fact that for the most part different spectral lines are used in the two methods, and the direct reading method is better suited to frequent and periodic standardization. The data given in Table I illustrate the precision obtained with the direct reading spectrometer under routine operating conditions in terms of the standard deviation* in per cent of the amount present, calculated from 200 runs of the standard sample used for the hourly standardization.

Table I — Precision of Analytical Results *(a)*

Element	Direct Reading Method			Spectrographic Method	
	No. 1	No. 2	No. 3	No. 4	No. 5
Aluminum at 4.4%	1.8	2.9	2.1	5.3	2.7
Calcium at 0.072%	2.5	3.5	2.8	5.9	
Zinc at 2.2%	1.5	1.9	1.6	3.7	3.6
Copper at 0.023%	3.7	7.3	4.2	6.8	5.7
Manganese at 0.18%	2.4	5.0	2.8	3.3	4.5
Silicon at 0.063%	4.4	11.4	6.7	10.4	
Beryllium at 0.0009%	7.2	12.4	8.3	10.9	

(a) Standard deviations for single determinations in per cent of the amount present, or A.S.T.M. "coefficient of variation" *v*. (See text.)

Column No. 1 — "Instantaneous" accuracy, not dependent upon instrumental drift.
No. 2 — Standard deviation for 200 runs, related to stability of apparatus over 100 hr.
No. 3 — Standard deviation for operating conditions; periodic standardization.
No. 4 — Standard deviation for 44 runs.
No. 5 — Standard deviation reported by others in the literature.

In column No. 1 the "instantaneous" accuracy has been calculated from the difference between the two determinations made each hour according to the formula

$$\sigma = \left(\frac{\Sigma d^2}{2N}\right)^{1/2}$$

where d is the difference occurring within a pair and N is the number of pairs. This calculation gives a figure which depends only upon the stability of the apparatus for the length of time required to run duplicate determinations. Column No. 2 gives standard deviations calculated from the same set of 200 runs according to the usual formula

$$\sigma = \left(\frac{\Sigma x^2}{n}\right)^{1/2}$$

where x is the deviation of each run from the arithmetical mean and n is the total number of runs; these figures therefore are related to the stability of the apparatus over a period of 100 hr.

*See the A.S.T.M. "Manual on Presentation of Data", 1941.

assuming no standardization is made during the entire period. Actual operation is carried out under conditions between these extremes, since periodic standardization reduces to a considerable extent the effect of instrumental drift.

The figures in column No. 3, which are standard deviations for operating conditions, were calculated by combining the figures of column No. 1 with the root-mean-square hourly variation in the standardization figure. If the routine samples are run in duplicate, the precision would be somewhat better than indicated here.

For comparison with the direct reading method, the same sample used as described for periodic standardization was run photographically 44 times, each on a separate plate. The standard deviations obtained from these data are given in column No. 4. This laboratory as well as others* have reported precision data on the photographic spectrochemical analysis of magnesium alloys, some of which are more favorable than indicated by column No. 4; results reported previously in the literature are listed in column No. 5.

It is evident from the data given in the table that the direct reading spectrometer in its present form is capable of duplicating the analytical precision of the best photographic procedures. After several years of development and application of direct reading methods, it seems certain that accuracy far superior to that of the photographic method will often be achieved. At present, in the more favorable elements, the new method has approximately twice the accuracy of the old.

Tests with constant light sources — such as a tungsten lamp operated from batteries — have shown that the spectrometer itself can measure relative intensities with better accuracy than 0.5%. The largest source of error in the assembly is therefore in the spark excitation unit (and perhaps the samples themselves). Any major improvement in the accuracy of the instrument as a whole will thus have to come from improved sampling techniques and better spark excitation.

*T. M. Hess and L. G. Reinhardt, *Journal* of the Optical Society of America, V. 34, p. 104; B. L. Averbach, *Industrial and Engineering Chemistry*, Analytical Edition, V. 17, p. 341; K. E. Mann, *Spectrochimica Acta*, V. 1, p. 563; G. P. Carter, *The Metal Industry*, V. 60, p. 140.

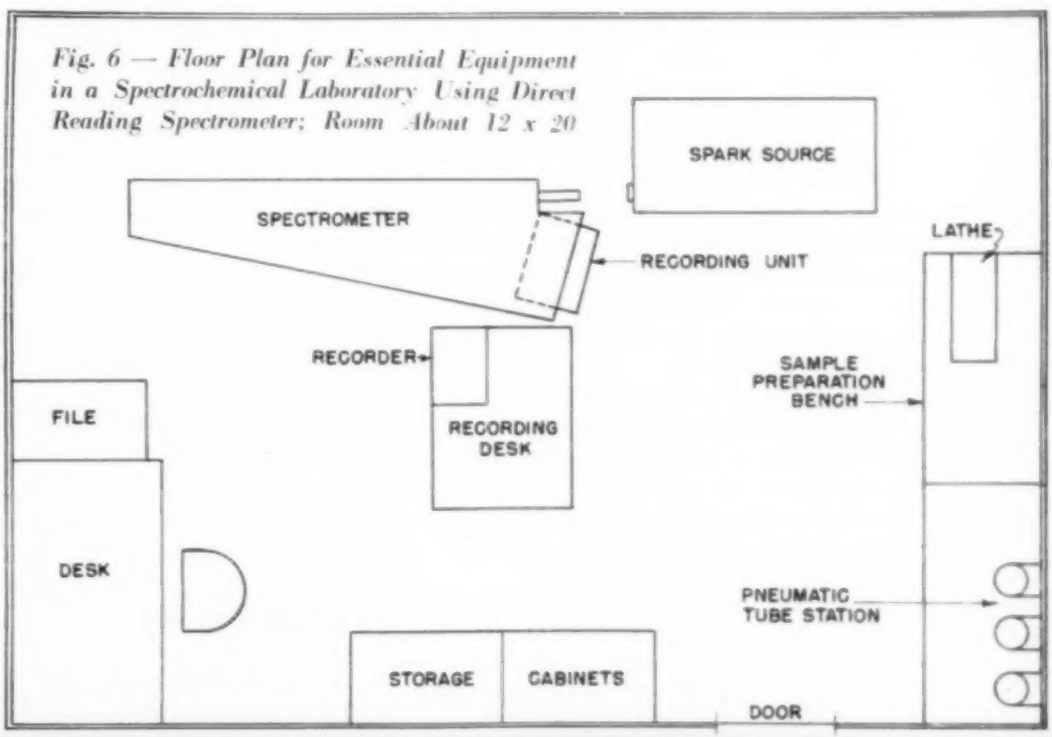
Speed — Rapidity of analysis is perhaps the most striking feature of the direct reading spectrometer. The importance of rapid methods is well illustrated by the development within recent years of well organized spectrographic laboratories designed to analyze samples taken from alloying pots during the manufacturing process. Speed in returning the analytical results to those in charge of the alloying process means shorter heats, better output per furnace, and more uniform metal.

A well organized team consisting of four or five operators, each trained for a specific job, can complete the analysis of a sample for several elements with the photographic method within 10 min. Over a period of 8 hr. an average time of 15 min. per sample would be considered good. Frequently this rate is impossible because one of the expert operators may be absent, necessitating a substitute on an unfamiliar job.

On the other hand, two operators with a direct reading spectrometer can maintain a pace of one sample every 5 min. without being rushed to the extent that numerous careless errors are made. Indeed, there are considerably fewer places where mistakes *can* be made. It is feasible to run duplicate determinations on each sample for additional accuracy with a negligible increase in operating costs. In general, the new method is from three to six times faster than the photographic, and less effort is required on the part of the operators to maintain this pace.

Cost of Operations — One well-known spectrographic laboratory making analyses by the photographic method for production control reports using a five-man crew (a total of 15 for three shifts) to handle about 150 samples a day (24 hr.). This laboratory uses about ten dozen plates a day. Daily operating costs for men and

Fig. 6 — Floor Plan for Essential Equipment in a Spectrochemical Laboratory Using Direct Reading Spectrometer; Room About 12 x 20



materials would be about $180. Analytical costs are thus about $1 per sample, or 20¢ per determination on the basis of five per sample.

With the direct reading spectrometer one operator per shift (three for 24 hr.) could handle the same load. Use of a mechanical computer, in place of the recorder described earlier which uses electrical marking paper, would thus make the daily operating expense of the laboratory less than $45. In addition, the direct reading spectrometer will need a certain amount of servicing, but on the basis of a periodic check-up every three months by a service engineer representing the manufacturer of the equipment, as well as a complete set of new tubes every six months, one might expect to spend $1000 per year in this manner. Even so, the cost of operating the direct reading spectrochemical laboratory would be on the order of one-fourth as much as that required to perform the same amount of work photographically. One direct reading spectrometer will not handle a great many more samples per hour than will a complete photographic laboratory; the saving in costs comes from the use of fewer operators.

Skill of Operators — In the photographic spectrochemical laboratory the routine operator must be taught a variety of duties among which are the preparation of the sample, the operation of the spectrograph, the proper photographic processing technique, the use of the microphotometer or densitometer, and the operation of the calculating board. In addition, he may be expected to calibrate the plate or film and to be familiar with and recognize the various spectral lines used. The pitfalls threatening the inexperienced operator are many, and frequent errors are to be expected even of the more experienced workers.

In contrast to this situation, the operation of the direct reading spectrometer is entirely automatic except for the preparation of the electrodes and getting them into the clamps in the proper position for sparking. Standardization is direct and simple and frequent checks can readily be made. It is no exaggeration to say that operation of the direct reading spectrometer can be taught in a day, although of course familiarity with the customs and routine of the laboratory would require longer. Since the spectrometer can be safely used overnight without any adjustments whatever, reliable results can be obtained with a minimum of skill.

Sensitivity — Accuracy for trace elements, or sensitivity of the direct reading spectrometer, appears to be of the same order as that obtainable by photographic methods. The spectrometer described here has somewhat better accuracy on elements near the limit of detection than the routine spectrographic method used in Dow Chemical Co.'s laboratory. However, a valid comparison would require the use of the same spectral dispersing system for both methods. J. L. Saunderson, V. J. Caldecourt, and E. W. Peterson of this laboratory have reported the use of the direct reading spectrometer for the determination of beryllium in magnesium alloys at a concentration of 0.0001%. A standard deviation of 0.000,02% was obtained, indicating that one part per million can be distinguished from two parts per million. G. H. Dieke of Johns Hopkins University also reported the detection of silicon in steel at a concentration of 0.01% by the scanning method described below.

Applications of Direct Reading Instruments

The possibility of replacing the photographic plate with a photoelectric device of some sort has intrigued spectrographers for some time. One of the first successful attempts was made by Duffendack and Morris,* who used photosensitive Geiger-Müller photon counters mounted on a small quartz prism spectrograph. Some encouraging results were reported, but the difficulties encountered in making reliable counters for operation at wave lengths of light longer than 2600 Å has no doubt limited further work with these counters. Rank, Pfister, and Coleman† quickly realized the usefulness of the newly developed electron multiplier phototube in the field of spectroscopy, and worked out a successful method for photoelectric recording of Raman spectra.

More recently, Boettner and Brewington‡ used a small grating spectrograph with two multiplier tubes arranged in a bridge circuit, thus determining one element at a time by manually balancing the bridge. Hasler and Dietert¶ published a paper based upon some preliminary data obtained with a direct reading photoelectric instrument called the Quantometer, also using electron multiplier tubes. Dieke** obtained considerable information on the properties of electron multiplier tubes, and discussed briefly a scanning method of quantitative analysis.

There are at present three types of equipment

*O. S. Duffendack and W. E. Morris, *Journal* of the Optical Society of America, V. 32, p. 8.

†D. H. Rank, R. J. Pfister, and P. D. Coleman, *Journal* of the Optical Society of America, V. 32, p. 390.

‡E. A. Boettner and G. P. Brewington, *Journal* of the Optical Society of America, V. 34, p. 6.

¶M. F. Hasler and H. W. Dietert, *Journal* of the Optical Society of America, V. 34, p. 751.

**G. H. Dieke, *Journal* of the Optical Society of America, V. 33, p. 421.

proposed for the direct spectrochemical method. These three types might be called the "fixed slit" the "movable receiver", and the "scanning" method. As will be apparent, each method has its own inherent advantages and limitations.

The fixed slit method, as described in this paper, is generally applicable to situations where the analytical job has been reduced to a routine —that is to say, many analyses are required on the same type of material for perhaps a dozen elements. Under these circumstances, the necessary equipment is rugged, is simple to operate, requires a minimum of adjustment, and can readily be attended by non-technical help. This method has the advantage that the fluctuations normally occurring in a spark source are integrated or averaged out over an appreciable period, perhaps 20 sec. It is less expensive than the movable receiver type. Furthermore, in the fixed slit spectrometer a great deal of care is taken in originally setting up the instrument to get all the exit slits into exact alignment. Exit slits for all the elements for which an analysis is desired should preferably be located properly during the initial installation, inasmuch as the slits are not readily movable from one spectral line to another. Thus the fixed slit spectrometer lacks adaptability, and it can be used only on the type of material for which it was originally installed. There is, however, the possibility that these limitations could be relaxed somewhat. For example, the common elements used for alloying with both magnesium and aluminum are the same in most alloys, so that a fixed slit spectrometer could probably be made to handle both families of alloys.

The movable receiver type of instrument, as exemplified by the Quantometer, is constructed so that the multiplier tubes can be moved along the focal curve of the spectrometer by controls extending outside the instrument. This method has more adaptability than the fixed slit method and probably will be most useful for research laboratories encountering a variety of problems. It would also be useful in developing the routine control methods to be used with the fixed slit spectrometers located in outlying plants and foundries. More experienced spectroscopists would be required to make full use of equipment of this type.

The scanning method as proposed by Dicke has the widest applicability of the three methods but probably the poorest accuracy in its present state of development. It uses one or more multiplier tubes moving along the focal curve during the spark exposure, and a device for recording the variations in light intensity. The record is similar in appearance to a recording microphotometer trace of a spectrum taken on a photographic plate. With this method there is no limitation of the spectral lines which may be used and hence any analysis which can be done by photographic spectrochemical methods can be attempted by this direct recording method. However, the scanning method rules out the possibility of averaging the light intensities of the spectral lines over a period of several seconds. Inferior accuracy is therefore to be expected until further development of spectral sources can give a much more stable excitation of the spark.

It is not the purpose of this paper to imply that the photographic method is "on the way out", but rather that these direct reading methods will supplement the photographic, and often do a much better job—particularly of a routine nature. The combination of a fixed slit spectrometer and the photographic method has proved very successful in Dow Chemical Co.'s laboratory; the spectrometer handles the routine alloy samples and the spectrograph the more unusual and varied analytical problems. Whether such a combination of methods is eventually replaced by a direct reading method of the scanning type remains to be seen.

Within a year or two direct reading photoelectric spectrometers of all three types will probably be available commercially. As has been suggested here, probably each type will be found useful for various kinds of analytical work. The manufacturers of this equipment will maintain a staff of field engineers, available on call, to service the equipment in emergencies, and perhaps on a periodic basis. The reliability of the equipment will be increased by having replacement units, amplifier assemblies, and power supplies on the shelf. Direct reading equipment may eventually have as great an influence upon industrial methods as did the spectrographic method in replacing chemical analysis.

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3. Get the name of a French family from the staff of Metal Progress, 7301 Euclid Ave., Cleveland 3, and send them a food box monthly (see page 1075).

Metallography

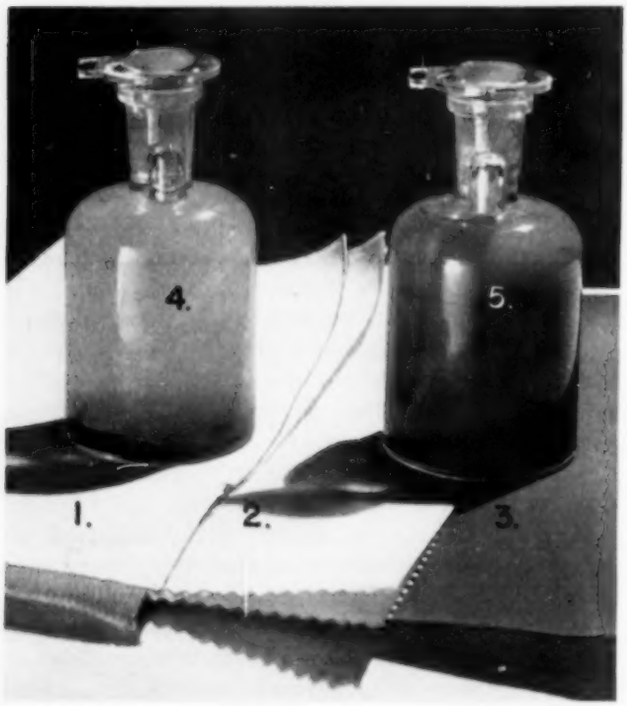
A Universal Metallographic Polishing Procedure

By William C. Coons
Supervisor, Metallographic Laboratory, Curtiss-Wright Corp., Propeller Division, Caldwell, N. J.

A UNIVERSAL polishing procedure capable of producing a finish that is highly satisfactory for metallographic examination or photography at any magnification and on any type of metallic material is the ideal envisioned by every metallographer. Its realization, however, has come to be regarded as wishful thinking, inasmuch as several common metals and alloys have so far required special polishing techniques.

From the time the microscope was adopted as a metallurgical tool, many kinds of polishing cloths, abrasives, and techniques have been fabricated, compounded, and proposed, each supposedly having extraordinary physical characteristics or other special advantages. Today, nonferrous metallographers advocate procedures that are frowned upon by those examining ferrous metals. However, there are many who are required to handle both ferrous and nonferrous materials, and it is they who are most keenly aware of the need for an all-purpose polishing procedure that can be depended upon to give truly excellent results on the greatest variety of metals and alloys.

Fig. 1 — Materials Used in Polishing Method



The procedure to be described is offered with the conviction that it satisfies these requirements. The photomicrographs of iron, steel, and alloys of aluminum, copper, and magnesium, are offered in support of this statement, and it is hoped that they will speak for themselves. With the method long practiced in the metallographic unit of the engineering materials laboratory of Curtiss-Wright Corp., metallographers of no greater than average ability have produced results comparable to those illustrated. All anyone need do is observe the precautions and follow the procedures which will now be outlined.

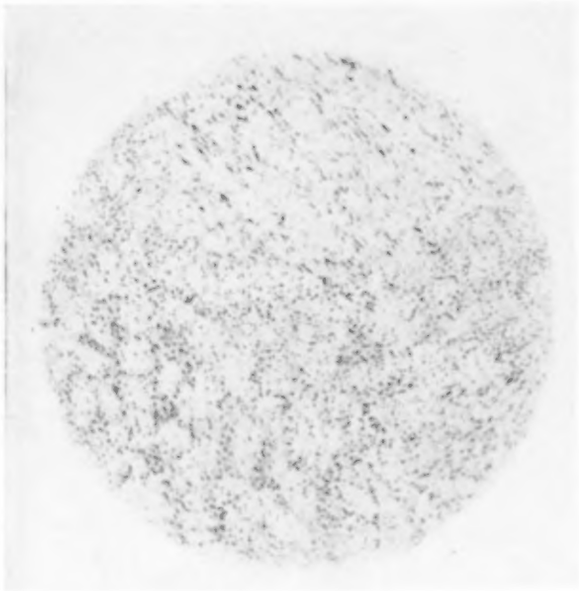
Essential Precautions — The secret of success in metallographic polishing resides in a few simple precautions which must be observed religiously by any metallographer, unless he is satisfied with mediocre or poor results. Since these precautions are the foundation without which any polishing technique will be predestined for failure, they will be enumerated first, and should be studied and remembered before the new universal method is given a trial.

1. Coarse abrasives should never be carried by the fingers or the specimen from one cloth to another. The hands should be washed and the specimen thoroughly swabbed with cotton under running water; then both hands and specimen should be dried in a hot air stream or on a clean cloth before proceeding to the next operation.

2. Polishing should not be prolonged in any

one direction. On the final cloths the specimen should be rotated clockwise against the counter-clockwise direction of the wheel (or twisted or oscillated by the fingers) and at the same time moved deliberately from the center toward the outer edge of the cloth-covered disk.

Fig. 2 — Alpha-Delta Eutectoid Plus Beta Plus $(Fe.Ni)Al_3$ in an Alpha Matrix in an Aluminum Bronze, Furnace Cooled From 1650° F. Rockwell B-70. Etched lightly in 1% chromic acid. 100×



3. Regardless of technique, polishing should not be prolonged. A metal being polished should be *cut* by the abrasive but not *rubbed* by the cloth. Therefore use enough abrasive that has not been diluted excessively.

4. Water should not be allowed to dry on the polished surface after the termination of one operation and before starting the next. After cleansing the abrasive material from the specimen thoroughly rinse with alcohol and dry in a stream of warm air.

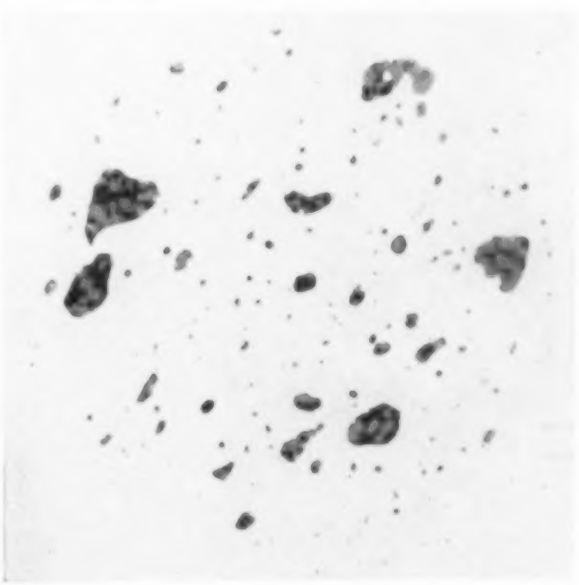
Fig. 3 — Same as Fig. 2. 1000×

5. After securing the cloths on the wheels, make sure that sufficient excess is trimmed off the edges to prevent used abrasive material on the sides or bottom of the wheel-guard from being splashed back onto the cloth.

Disregard of these precautions will cause coarse scratches, will drag out inclusions with associated "fishtailing", and will pit the specimen, all defective conditions which will defeat the most ardent efforts to produce a good polish no matter what methods or materials are employed.

Materials — The cloths and abrasives employed by the new method are illustrated in Fig. 1. There are three cloths, namely, canvas (1), silk (2), and "Gamal" (3). For best results the silk cloth should be soaked for at least 24 hr. in a 2% solution of hydrochloric acid and then, prior to being placed into operation, washed with tincture of green soap and rinsed thoroughly in distilled water. If it is contaminated by foreign abrasive particles the cloth may be removed and washed.

Fig. 4 — Transverse Section of Wrought Iron Showing Slag Inclusions. Rockwell B-56. Unetched. 400×



The abrasive employed on the canvas cloth is 600-mesh Carborundum mixed with distilled water in a ratio of 1:10 (bottle 4 of Fig. 1).

A polishing abrasive called "C-RO"* has been found superior to all others for use on both the silk and "Gamal" cloths. This compound also prevents the cloths from drying out over reasonable periods of idleness. A dilution of one part of "C-RO" to no more than three parts of distilled water is recom-

*"C-RO" is Manufactured by The Marwin Co., 12 Distler Ave., West Caldwell, N. J.

Fig. 5 — Ferrite and Pearlite in Normalized 1020. Rockwell B-74. Etchant: 4% picral and 2% nital. 100×

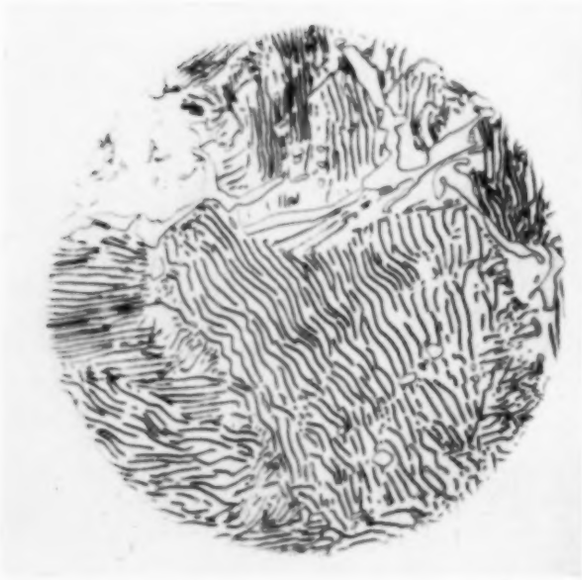


Fig. 6 — Pearlite and Excess Cementite in S.A.E. 1095, Furnace Cooled From 1650° F. Rockwell B-87. Etched in 4% picral. 1000×

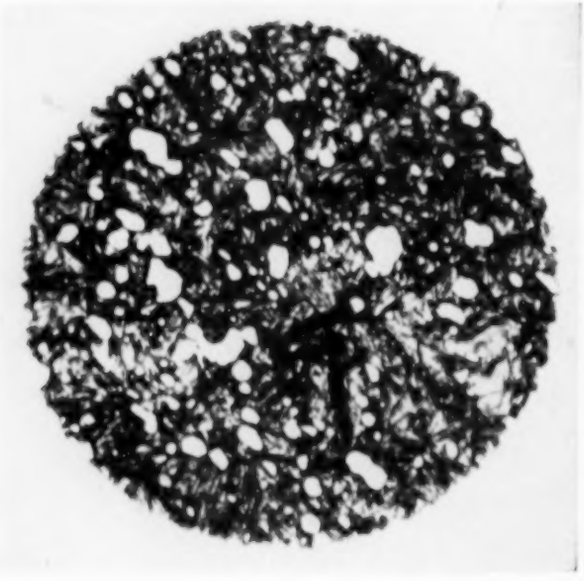


Fig. 7 — Martensite and Carbides in Toolsteel. Rockwell C-66. Etched with Vilella's reagent. 1000×

mended (bottle 5). Even though only a few drops are applied to the cloth, the polishing time is amazingly short and the resulting polished surface is highly satisfactory.

Procedure — For illustrative purposes the step-by-step procedure used to polish a specimen of aluminum bronze is described. The sample had been furnace cooled from 1650° F.; its hardness was Rockwell B-70.

Step 1: Grind, preparatory to cloth polishing, on coarse and fine vertical abrasive wheels and on numbers 1, 0, 00, and 000 metallographic emery paper. (This operation may be performed in any other accepted manner.)

Step 2: Rough polish on the canvas cloth wheel, rotating at a speed of 240 r.p.m. In order to insure good cutting, apply the 600-mesh Carborundum from a dropping bottle during the polishing. Reverse the direction of polishing every few seconds by lifting the specimen from the wheel, turning 180° and placing it again in contact with the cloth and abrasive; continue until all of the emery paper scratches are removed. Thoroughly wash and dry the specimen and hands.

Step 3: Place a few drops of "C-RO" on the silk cloth from a dropping bottle. Using a wheel speed of 550 r.p.m., polish by rotating the specimen opposite to the direction of the revolving cloth, utilizing only the area near the center. Apply medium pressure; when gripping of the specimen commences, add a few more drops of abrasive. To determine when the scratches produced by the previous polishing operation are removed, wipe the surface of the specimen clean with a piece of facial tissue. Finally, wash and dry specimen and hands.

Whether hard or soft, most specimens can be examined for inclusions or structure after this operation. However, so-called research specimens and those to be photographed should be finish polished on the "Gamal" cloth.

Step 4: Add "C-RO" abrasive to the "Gamal" cloth as in Step 3. The technique of polishing found to produce the best results is to hold the sample under light pressure near the center of the wheel (speed 550 r.p.m.) and then with increased pressure and a twisting motion by the fingers, move it toward the outer portion of the cloth. When the fingers can twist no farther the specimen is removed and the motion repeated. (When very soft specimens are being pre-

pared, it is advantageous to finish polishing with a light buff, using a wheel speed of about 1150 r.p.m.) Cleanse the specimen from abrasive by swabbing it with cotton under running water, wetting with ethyl alcohol, and drying in a stream of warm air.

Fig. 8 — Silicon (Gray) and Al-Cu-Fe-Mn (Black) in Aluminum Alloy No. 43 (As Cast). Hardness below Rockwell B scale. Etched in Keller's reagent. 500×

Results — The time required to polish this specimen of aluminum bronze on the canvas, silk, and "Gamal" cloths was 2 min. Photomicrographs, Fig. 2 and 3, illustrate the excellent polish attained in this short time and without the customary re-buffing and duplicate etching.

All specimens handled in Curtiss-Wright's Propeller Division laboratory have been prepared as described above (including Step 1). Figures 4 through 10 are offered as additional proof that the recommended cloths, abrasives, and techniques really can be considered to constitute a truly universal hand polishing method. The specimens from which the photomicrographs were taken were polished and etched only once, the maximum polishing time being 3½ min. — this on the magnesium alloy specimen.

Fig. 9 — Coring in 85-15 Copper - Manganese (As Cast). Rockwell B-29. Etched in 1% chromic acid electrolytically. 150×

Summary — The universal metallographic polishing technique which has been described in the preceding paragraphs may be summarized as follows:

1. The polishing cloths are canvas, silk, and "Gamal" (1, 2 and 3, Fig. 1).

2. The abrasives are 600-mesh Carborundum and "C-RO" (4 and 5, Fig. 1).

3. Techniques are largely personal but the ones described are those found to be most satisfactory.

4. Any type of metal or alloy encountered could be polished by the same procedure in 2 to 4 min.

5. In most instances, even specimens for high power research were prepared with one polish and etch for photomicrography at any magnification. The success of this simplified procedure is attributed to the effective manner in which the abrasives, not excessively diluted, cut away the disturbed or flowed metal produced by the grinding operations.

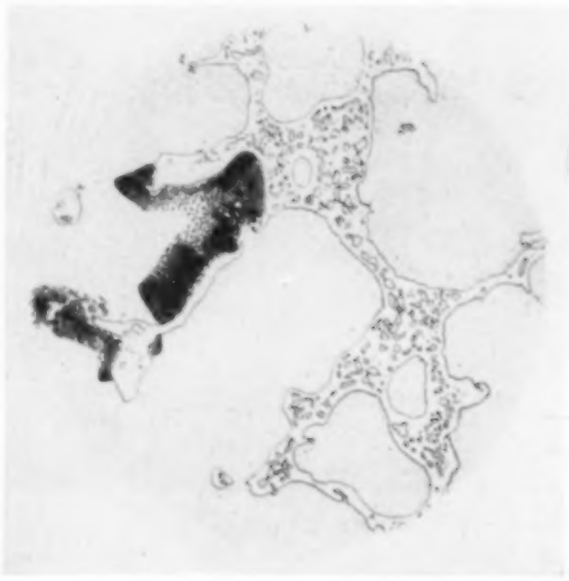


Fig. 10 — B (Mg-Al) Eutectic and Lamellar B (Mg-Al) in Magnesium Solid Solution in Magnesium Alloy (As Cast). Rockwell B-30. Etched in glycol etchant for 10 sec. 500×

In conclusion, the results we have attained with these techniques and materials have exceeded the most optimistic expectations, and the new method is passed on as a contribution to the development of the ultimate, universal polishing procedure.

Observations on the Impact Strength of Alloy Steels Hardened From Cyanide Baths

By Sidney M. Lenhoff and Louis F. Roth
Metallurgists, Detroit Diesel Engine Division of General Motors Corp. and Deere & Co., Respectively

ONE of the most interesting points of discussion in the metallurgical field today is the role that nitrogen plays in steel. It is agreed that it is mainly responsible for the high hardness obtained in cyanided steel parts. On the other hand there is evidence that it is detrimental in lowering impact resistance to a marked degree. Our interest in the latter phase of this subject was first aroused when complaints were received from the engine test room at our plant that rollers were failing.

Initial inspection of these parts, which are made from S.A.E. 52100 steel, "ball bearing steel", showed a very brittle looking fracture. This seemed peculiar, because the impact load on this part in service is very low. Standard heat treating practice for these rollers, which were manufactured and heat treated by an outside supplier, consisted of heating in cyanide at 1550° F. for 15 min., quenching in oil and drawing for 3 hr. at 350° F. Blueprint specifications called for a hardness of Rockwell C-60 minimum.

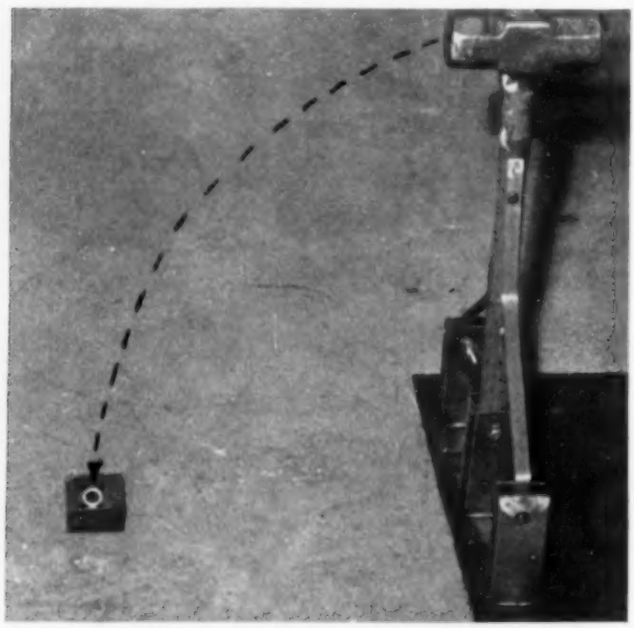


Fig. 1 — Preliminary Fixture (6-Lb. Hammer) for Testing Unsatisfactory Rollers Used in Diesel Engines

The situation could have assumed serious proportions and an answer was needed immediately. We therefore rigged up the crude fixture shown in Fig. 1 wherein a 6-lb. sledge hammer swung through an 18-in. arc. A few tests gave an answer sufficient to indicate the need for a better heat treatment. The preliminary results on a sampling of a shipment of these rollers showed them to meet the hardness specification (C-60 to 61) but they broke under from 2 to 31 blows, a large scatter that averaged 9.5.

Various heat treatments were then tried to determine the effect of time, temperature and heating medium upon the toughness of these rollers. Lots were heated in cyanide and in a controlled atmosphere furnace (Lindberg "Hydryzer") for various times and at different temperatures and then tested in the fixture. All samples were oil quenched and drawn at 325° F. for 1 hr. Results are shown in Table I.

These results indicated that atmosphere controlled heating gave better toughness than heating in cyanide. It may be noted that the results obtained on our cyanide treatments (1 to 5 blows to fracture) were much worse than the 2 to 31 obtained when testing the parts which had been cyanided at the outside source. We thought the difference might be due to the fact that the cyanide bath of the outside vendor contained only 10% NaCN, while the one used in our tests contained approximately 20% NaCN.

Our attention then turned to hot oil quenching as a possible way to improve the properties. Following along this line five treatments were

investigated. Since the experiments of Table I indicated that 1550° F. was too high a quench, the hardening temperature was reduced to 1475° F. A summary of the results is contained in Table II. As a result of these tests we have adopted treatment 1-B as production practice and since that time we have received no further reports of trouble.

Although our problem of unsatisfactory rollers was solved by changing the heating medium and the quenching practice, it left some insistent questions in our mind as to why cyanide should act in this way. Some further investigation was warranted, since

Table I — Hammer Blows to Fracture 52100 Rollers
(All oil quenched and drawn at 325° F. for 1 hr.)

Heat Treatment			Toughness		Hardness
Medium	Temp.	Time	Range	Average	
Cyanide	1550° F.	10 min.	4 to 5	4.3	C-62
Cyanide	1550	30 min.	2 to 3	2.2	62.5
Cyanide	1550	45 min.	2 to 5	2.6	62
Cyanide	1550	1 hr.	1 to 3	1.9	62
Cyanide	1550	2 hr.	1 to 4	2.1	62
Cyanide	1550	3 hr.	1 to 4	2.2	62.5
Cyanide	1550	4 hr.	2 to 5	2.8	62
Hydryzer	1550	20 min.	4 to 25	12.2	62
Hydryzer	1450	20 min.	59 to 70	65.3	62.5
Cyanide	1450	20 min.	1 to 27	11.8	59.5

Table II — Results of Hot Oil Quenching 52100 Rollers, Austenized at 1475° F.

Treatment No.	Heating Practice			Quench		Draw		Range of Blows	Core Hardness
	Medium	Temp.	Time	Medium	Time	Temp.	Time		
1-A	Hydryzer	1475° F.	30 min.	Oil at 200° F.	30 min.	325° F.	1 hr.	5 to 56	C-60 to 61
1-B	Hydryzer	1475	30 min.	Oil at 250	30 min.	325	1 hr.	50 to 100+	60 to 61
1-C	Hydryzer	1475	30 min.	Oil at 300	30 min.	325	1 hr.	18 to 96	60 to 62
1-D	Cyanide	1475	20 min.	Oil at 250	30 min.	325	1 hr.	15 to 40	55 to 59
1-E	Cyanide	1475	10 min.	Note *(a)*		325	1 hr.	10 to 26	60 to 61

Note *(a)* — Cold oil for 9 sec., then oil at 250° F. for 30 min.

cyanided parts have given superior performance where *wear* is the prime consideration. We wanted to know what to do with a part that needed toughness as well as wear resistance.

It was therefore decided to run similar tests on "tips" for that same engine. These are machined from S.A.E. 6140 or 6145 steel, and hardened from cyanide. For this purpose we designed a machine with a swinging hammer similar in nature to that used on our preliminary work. Figures 2 and 3 show the machine and the sectioned part, together with the fixture which enables the hammer to strike against the barrel of the tip.

Before discussing the results of the treatments given the tips, it should be mentioned that this part is such as to offer a definite notch effect (see Fig. 3). The arrow denotes the point of impact. Because of the thin wall and the inevitable difference in the machining of the step on the inside diameter, we obtained some fluctuation in results. However, a sufficient number of each lot were run to insure an accurate indication of the effect of each heat treatment.

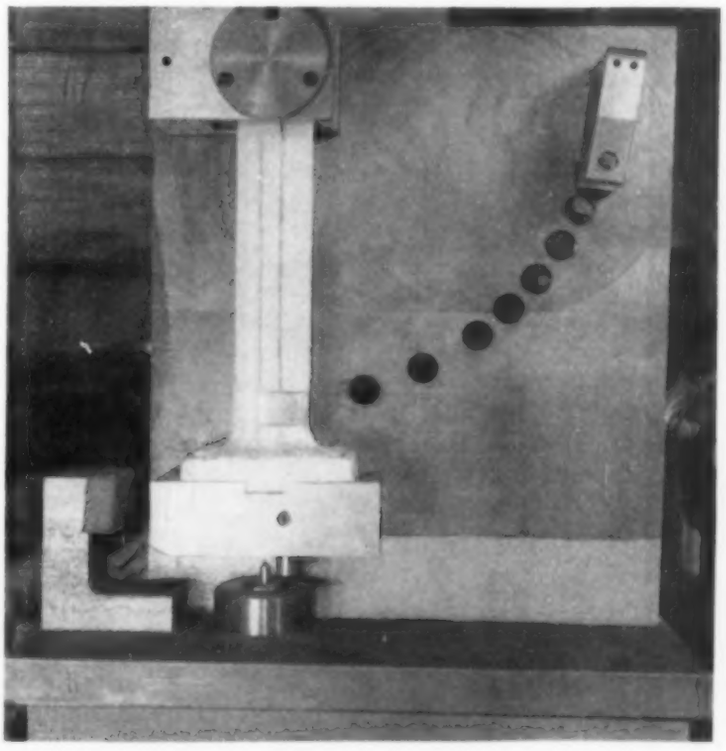


Fig. 2 — Impact Tester Is a Swinging Hammer, Similar to the Izod or Charpy Machine. Tip under test is held in round fixture attached to machine base; hammer is shown in position where it is about to strike the tip. Number of blows to fracture, from 80° swing, is measure of toughness of sample

Table III — Toughness and Hardness of 6145 Tips Heat Treated in Various Ways

Treatment No.	Heating Practice			Quench		Draw		Range of Blows	Core Hardness
	Medium	Temp.	Time	Medium	Time	Temp.	Time		
2-A	Cyanide	1540° F.	45 min.	Oil at 80° F.	——	550° F.	1 hr.	2 to 15	48 to 49
2-B	Cyanide	1540	45 min.	Salt at 525	45 min.	550	1 hr.	2 to 12	48.5 to 49.5
2-C	Cyanide	1540	45 min.	Salt at 525	1½ hr.	550	1 hr.	1 to 6	48 to 50
2-D	Cyanide	1540	45 min.	Salt at 525	2½ hr.	550	1 hr.	1 to 4	47.5 to 48.5
2-E	Cyanide	1540	45 min.	Salt at 525	3½ hr.	550	1 hr.	1 to 4	47.5 to 48.5
2-F	Hydryzer	1540	45 min.	Oil at 80	——	650	1 hr.	12 to 19	48 to 48.5
2-G	Hydryzer	1540	45 min.	Salt at 475	45 min.	650	1 hr.	21 to 34	48 to 48.5
2-H	Hydryzer	1540	45 min.	Salt at 475	1½ hr.	650	1 hr.	26 to 76	48 to 48.5
2-I	Hydryzer	1540	45 min.	Salt at 300	1½ hr.	650	1 hr.	15 to 100+	49 to 52
2-J	*(a)*	1575	20 min.	Salt at 525	45 min.	650	1 hr.	3 to 9	47 to 48

Note *(a)* — Liquid carburizing bath.

A comparison of the results shown in Table III indicates that cyanide treatments 2-B, 2-C, 2-D, and 2-E cause a decline of toughness with increasing time at 525° F. Microstructural examination of the respective samples showed that austenite is retained tenaciously; apparently the cyanide treatment had increased the stability of the austenite to such an extent that 3½ hr. were required for complete transformation of the case. The longer the tempering time the less the retained austenite and the lower the toughness — although none of the pieces was exactly what could be called "tough".

The results obtained on repeated impact test would seem to be conclusive, and checked the findings on rollers, and so justified our impression that cyanide hardened alloy steels were rather brittle. However, the point was raised that the test was unfair due to the fact that the cyanided tips had a "case" (a casehardened surface) whereas samples 2-F to 2-I had none. It was further stated that perhaps it was the carbon penetration (carbon derived from the cyanide bath) which was giving the poor results. Note that treatment 2-J, hardening from a liquid carburizing bath, gave tips that fractured with relatively few blows as compared to the tips hardened from the Hydryzer atmosphere.

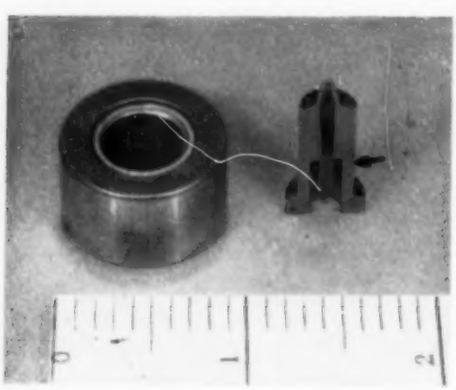


Fig. 3 — Roller Used in Diesel Engine and Sectioned Tip

Toughness of a Carburized Part

To study the effect of a carburized surface on the resistance of the tips to repeated hammer blows, we obtained more of the tips (S.A.E. 6145 steel, as before) and gave three lots of them the following treatments:

Treatment 3-A — Pack carburize at 1675° F., 5 hr. total cycle, cool in pot. (Case depth: 0.035 in.) Reheat in Hydryzer for 45 min. at 1550° F. and oil quench. Draw 1 hr. at 650° F.

Treatment 3-B — Pack carburize at 1675° F., 5 hr. total cycle, cool in pot. Reheat in Hydryzer for 45 min. at 1475° F. and oil quench. Draw 1 hr. at 700° F.

Treatment 3-C — Heat in gas carburizing furnace at 1675° F. for 2 hr. and air cool. (Case depth: 0.014 in.) Reheat in Hydryzer at 1550° F. for 1 hr. and oil quench. Draw 1 hr. at 650° F.

Subsequent testing in the fixture shown in Fig. 2 gave the results listed in Table IV.

These results show that carbon penetration certainly does not lower the toughness of the steel. In fact, these carburized tips were the toughest of all. One of the tips which was given treatment 3-A was replaced in the impact machine after surviving 100 blows, and the test continued to see just how many blows it would take. Failure finally occurred after 226 blows. When one compares this result to the resistance of even the best of the cyanided tips (treatments 2-A and 2-B of Table III), it becomes evident that a cyanide bath is an inferior way to harden.

These figures were shown to a representative of one of the largest manufacturers of salt baths. He was rather skeptical of our results and so, at his request, we gave him 50 tips to be heat treated in any manner he desired. They were then returned to us and tested in the impact machine. We quote the heat treatment schedules from his covering letter:

Treatment A — Heated 45 min. in a cyanide bath containing 25.19% NaCN, transferred to a neutral salt bath at 1540° F.

for 30 sec., quenched into salt at 455° F. for 2 min. and air cooled. The tips were then drawn for 30 min. in a bath at 645° F.

TREATMENT B — Heated 10 min. in a 28.8% NaCN bath, transferred to a neutral salt bath at 1540° F. for 30 sec., then quenched into a bath at 545° F. for 25 min.

TREATMENT C — Heated 45 min. in a 28.8% NaCN bath, transferred to a neutral bath at 1540° F. for 30 sec., then quenched into a bath at 545° F. for 25 min.

TREATMENT D — Heated 30 min. in a carburizing salt bath at 1700° F., transferred to a neutral salt bath at 1540° F. for 1 min., then quenched into a bath at 600° F. for 25 min.

TREATMENT E — Heated 30 min. in a carburizing salt bath at 1700° F., transferred to a neutral salt bath at 1540° F. for 1 min., oil quenched and then drawn in a bath at 650° F. for 30 min.

TREATMENT F — Heated 10 min. in a 30% NaCN bath at 1540° F., oil quenched and then drawn in a bath at 420° F. for 30 min.

TREATMENT G — Heated 45 min. in a 30% NaCN bath at 1540° F., oil quenched and drawn in a bath at 650° F. for 30 min.

Table IV — Toughness of Carburized Tips (6145)

TREATMENT	CORE HARDNESS	RANGE OF IMPACTS
3-A	C-50 to 52	100+
3-B	51 to 52	75 to 100+
3-C	47	100+

Hardnesses of these parts are shown in Table V, as well as the results of our repeated impact tests. Although treatment D (austempering from a carburizing salt bath) gave parts that on the average withstood 20 blows, some failed after a very few blows.

We notified the salt bath manufacturer of these results, and we quote a sentence from the letter sent in reply:

"The fact that all treatments involving 10 min. in cyanide yielded better impact than corresponding treatments involving 45 min. cyaniding must lead to the conclusion that the cyanide case has poor impact."

It will be noted that the majority of the parts which had been quenched were drawn in the vicinity of 650° F. Data taken from "Metals & Alloys Data Book" by S. L. Hoyt give the impact properties of S.A.E. 6140, when drawn at various temperatures, as follows:

TEMPERATURE	IZOD IMPACT
400° F.	11 ft-lb.
500	8
600	8
700	10
800	12.5
900	19
1000	20

Since the lowest impact values in Table V were from samples drawn at about 650° F., we made some further tests, quenching the samples and then drawing at a temperature outside the 500 to 700° F. range. This was done to examine the possibility that cyanided samples might be extremely susceptible to damage in this temperature zone.

We therefore heated several tips in a bath containing 20% NaCN at 1540° F. for 45 min., quenched them in oil and drew them 1 hr. at 400° F. These tested C-51 to 52 hard and averaged 9 blows before fracture (range: 2 to 14). A comparison run was then made by heating in Hydryzer at 1540° F. for 45 min., quenching in oil, and drawing 1 hr. at 400° F. These had the same hardness (C-51 to 52), but averaged 39 blows before fracture (range: 12 to 65).

While the low draw after cyanide quench gives tougher parts than a draw at 650° F. (see also treatments F and G in Table V) there is still a marked difference in parts whose treatments are identical except for the *method* of heating.

Penetration of Nitrogen

An explanation of this embrittlement caused by heating in cyanide is rather difficult. It has been suggested that nitrogen penetrates into the core of these specimens and thus lowers resistance to impact. However, we know of no definite evi-

Table V — Properties of Tips of S.A.E. 6145, Hardened From Salt Baths at 1540° F.

CODE	TREATMENT			HAMMER BLOWS		HARDNESS	
	HEATING MEDIUM	QUENCH	DRAW	RANGE	AVERAGE	QUENCHED	DRAWN
A	Cyanide, then neutral	455°; 2 min.	645° F.	1 to 5	2.3	56 to 58	49 to 51
B	Cyanide, then neutral	545°; 25 min.	None	5 to 10	7.2	47.5 to 48	—
C	Cyanide, then neutral	545°; 25 min.	None	2 to 4	3.0	51 to 52	—
D	Carburizing, then neutral	600°; 25 min.	None	3 to 38	19.8	48 to 49	—
E	Carburizing, then neutral	Cold oil	650	1 to 5	2.2	62.5 to 63	50 to 51
F	Cyanide	Cold oil	420	3 to 6	4.0	53 to 54	50 to 51
G	Cyanide	Cold oil	650	1	1.0	61 to 62	49 to 51

dence that establishes this as a fact. F. D. Waterfall, of the Cassel Cyanide Co. of England, refers to this point in an article entitled "Case Hardening in Liquid Baths", published in *Iron Age* for June 27 and July 4, 1940. He writes:

"Sometimes it is found that mild steel parts lightly cased in cyanide at a relatively low temperature fracture easily and show a bright crystalline core. It has been said that nitrogen from the cyanide bath penetrates during treatment into the core of the piece and produces brittleness in the same way as embrittlement in pickling is produced by hydrogen. The reason for the brittleness is almost certainly related to the steel structure before treatment.

Table VI — Carbon and Nitrogen Penetration Into S.A.E. 1015 After 2½ Hr. in 50% NaCN Bath at Various Temperatures

Cut	1740° F.		1650° F.		1560° F.		1470° F.	
	%C	%N	%C	%N	%C	%N	%C	%N
No. 1	0.86	0.18	0.91	0.22	0.81	0.21	0.73	0.55
No. 2	0.86	0.05	0.78	0.21	0.65	0.11	0.26	0.17
No. 4	0.74	0.04	0.57	0.06	0.41	0.05	0.20	0.05

"From very many analyses, it has been shown that carbon penetrates further into the steel during cyanide carburizing than does nitrogen introduced at the same time, indicating that the nitrogen picked up during treatment in the cyanide remains in the case."

Mr. Waterfall substantiates this last statement by the analyses contained in Tables VI and VII, where the successive cuts are 0.003 in. deep. The samples cyanided were covered by a carbonaceous layer (usually graphite) to prevent excessive fuming and reduce cyanide consumption. Whether or not the nitrogen contained in the steel 0.012 in. below the surface (0.04% or more) is enough to cause brittleness may be questioned. We have some light to throw on this question a little further on in the present article.

Although Mr. Waterfall takes exception to the reported brittleness of the cyanide case when applied to low carbon steels, he does state that brittleness has occurred in alloy steels with higher carbon:

"The heating of parts such as gears made of oil hardening alloy steels in cyanide baths, so as to improve wear resistance by conferring a superficial hard case, is now widespread. If impact tests are conducted on notched pieces treated *(a)* in cyanide and *(b)* in the muffle, it will be found that the conferring of a hard case produces a marked drop in impact resistance. In one series of tests, muffle hardened steel of S.A.E. 3250 type showed 8 to 10 ft-lb. Izod impact, while similar pieces treated in the cyanide bath at an equivalent temperature gave only 3 ft-lb. The presence of the case in the root of the notch in the specimen appears to have an important effect. In view of the freedom from failure by shock of cyanided gears, where the notch effect is less than with an Izod specimen, it is probable that unnotched specimens would show less difference in impact resistance between muffle and cyanide hardening."

Nitrogen in High Speed Steel

ASM *Transactions* for 1945 contains an article entitled "Experiment on Sodium Cyaniding of High Speed Steel Prior to Hardening" by John McIntyre of International Business Machines Corp.,

Table VII — Carbon and Nitrogen Penetration Into S.A.E. 1015 After 2½ Hr. at 1740° F. in Cyanide Baths of Various Strengths

Cut	50% NaCN		40% NaCN		30% NaCN		20% NaCN		10% NaCN	
	%C	%N	%C	%N	%C	%N	%C	%N	%C	%N
No. 1	0.91	0.11	0.92	0.10	0.76	0.12	0.51	0.12	0.52	0.26
No. 2	0.80	0.08	0.78	0.07	0.66	0.08	0.54	0.10	0.41	0.23
No. 4	0.64	0.05	0.61	0.06	0.52	0.06	0.32	0.07	0.31	0.08

which describes a method wherein three types of high speed steels are given a cyanide treatment at 1450° F. for 1½ hr., and then hardened and tempered in accordance with normal practice. Hardness values of Rockwell C-68 to 70 were obtained throughout 1-in. round sections. The author attributed the extreme hardness to diffusion of nitrogen, both during the cyaniding and particularly during the high temperature heating necessary for hardening.

These points were very strenuously attacked by Stewart G. Fletcher, research associate at Massachusetts Institute of Technology, who wrote that the nitrogen content (0.018%) reported by the author was well within the realm of normal nitrogen content for high speed steel.

Our point in listing these opinions of Messrs. Waterfall, McIntyre and Fletcher is not one of criticism but merely to show the divergence of views now held on this subject. As mentioned above, Mr. McIntyre attributed his remarkable results to nitrogen penetration. To check this, we ran tests of a duplicate nature on a steel containing 5.75% W, 4.15% Mo, 4.00% Cr, 1.70% V,

and 0.82% C (which we thought was comparable in analysis to one of the steels used by him). In spite of repeated tests in which we varied the time and temperature in the cyanide bath, we were unable to get any hardness higher than C-65 to 66 in the center of ½x¾x6-in. tools.

This digression may be regarded as somewhat outside our main line of inquiry. We, however, present it as one of the side lines of our search for evidence about the effect of nitrogen supposedly diffused deep down from the surface.

Relative Properties of Case and Core

Mr. Waterfall attributes cyanide brittleness in low carbon steels to unrefined structures, and in higher carbon steels to the presence of a case at the root of the notch on Izod bars. Although our tests were conducted on 0.40 and 1.00% carbon steels and thus would not fit into the category of low carbon steels with unrefined structures, we would like to point out that both these higher carbon steels were fine-grained electric furnace steels containing fine carbide spheroids and ferrite. This is shown in the photomicrographs. As to the influence of case at the root of the notch, the parts made from S.A.E. 52100 and tested by us were round in shape, containing no notches whatsoever, but when heated in cyanide baths they still gave results inferior to those obtained when heated in a "neutral" atmosphere.

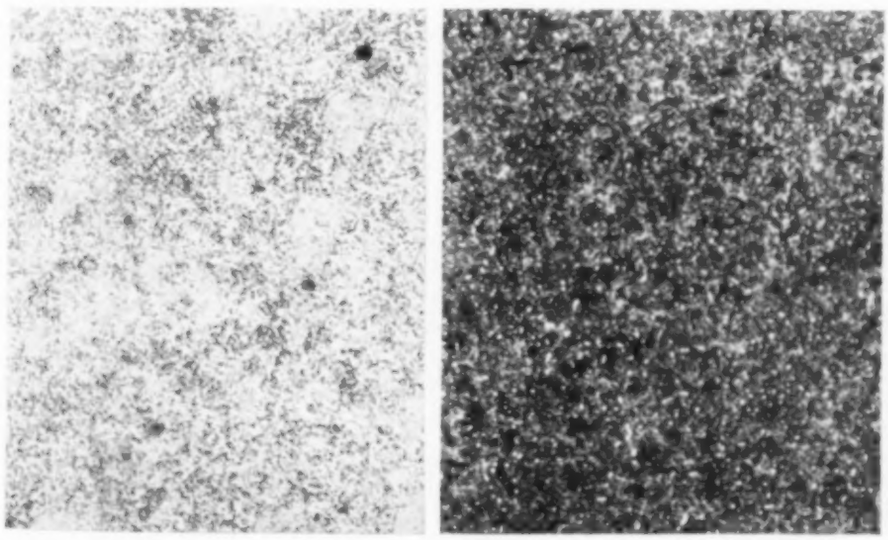


Fig. 4 and 5 — S.A.E. 52100 Used in Rollers. 500 ×. Left is structure before heat treatment. Right is the core after a heat in Hydryzer atmosphere at 1475° F. for 30 min., quench in oil at 250° F. for 30 min., and draw 1 hr. at 325° F.

However, the question of interest is whether or not nitrogen penetrates into the core of steels heated in cyanide baths. To determine this the following tests were run on S.A.E. 6140 steel: Eight samples were taken from one ⅝-in. round and machined into double-length square Izod bars.

TREATMENT 5-A — Machined and ground to Izod measurements; heated in Hydryzer at 1540° F. for 45 min., oil quenched and drawn 1 hr. at 400° F.

TREATMENT 5-B — Machined and ground to Izod measurements; heated in cyanide at 1540° F. for 45 min., oil quenched and drawn 1 hr. at 400° F.

TREATMENT 5-C — Machined and ground 0.020 in. over limits of test bar; no notch ground; heated in Hydryzer at 1540° F. for 45 min., oil quenched and drawn 1 hr. at 400° F., returned to tool room, where specimens were ground to size and the notch ground in.

TREATMENT 5-D — Machined and ground 0.020 in. over limits; no notch ground; heated in cyanide at 1540° F. for 45 min., oil quenched and drawn 1 hr. at 400° F., returned to tool room where specimens were ground to size and the notch ground in.

It will be observed that the only difference in the pair 5-A and 5-B, and the pair 5-C and 5-D, is that the first was heated in Hydryzer or controlled atmosphere, and the second in cyanide. Core hardness of all treated samples was C-51. Izod results (in ft-lb.) follow:

TREATMENT	BAR 1	BAR 2
5-A	16, 18	16, 18
5-B	7, 7	6, 7
5-C	17, 17	17, 18
5-D	12, 14	13, 14

Bars treated in cyanide and controlled atmosphere, and tested as heat treated (5-A and 5-B), showed the same relationships as in the other tests already described — namely, the cyanided bars were deficient in toughness. Furthermore, the values obtained on 5-C and 5-D (where the case was ground off and the notch ground into the heat treated bar) indicated that the *core* was definitely affected when the bar was heated in cyanide (5-D).

In an effort to explain this and also, at the same time, to settle the question of nitrogen penetration, we submitted samples of 5-C and 5-D to our chemistry laboratory for nitrogen analysis. Some careful work by Messrs. Uhl and Anderson,

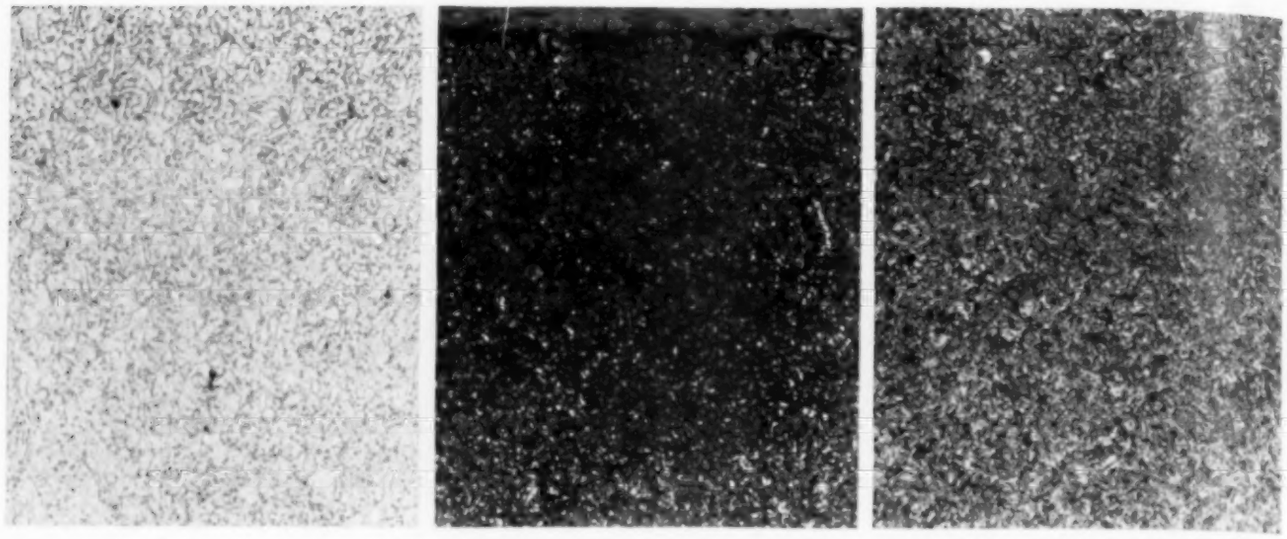


Fig. 6, 7 and 8 — S.A.E. 6140 Used in Tips. 500 ×. Left is structure before heat treatment. Center is case after heat in cyanide at 1540° F. for 45 min., oil quench and draw 1 hr. at 400° F. (Top edge of micro is mounting metal.) Right is core after same treatment

running parallel determinations in blank and against a Bureau of Standards steel certified to contain 0.024% N, was reported as follows:

Sample	Nitrogen Content
Standard	0.024%
Blank	0.000
Standard	0.024
5-C	0.0122
5-D	0.0122

Through the kindness of Messrs. Perso and Wills of Allegheny Ludlum Steel Corp., check analysis was made on the other halves of the Izod bars in their Dunkirk laboratory. This laboratory reported 0.010 for 5-C and 0.011 for 5-D. These results prove conclusively that whatever is the cause of brittleness in the core of cyanided parts, it is not the penetration of nitrogen.

Summary of Observations

1. Impact tests were run on S.A.E. 6140 and 52100 steels which had been austempered, quenched and drawn, or given interrupted hot oil quenches from cyanide, from a controlled atmosphere furnace, from molten carburizing salts, and after carburizing (solid or gas). Regardless of process, the samples heated in cyanide always gave the lowest results upon being tested for toughness.

2. The best results were obtained on S.A.E. 6140 by carburizing at 1675° F. in either solid or gaseous media, slow cooling, reheating in a controlled atmosphere, quenching in oil and drawing for 1 hr. in the vicinity of 650° F.

3. A minimum of 3½ hr. was required for complete transformation of the case of S.A.E. 6140 parts at 525° F. after austenizing at 1540° F. in cyanide, possibly due to stabilization of the austenite by nitrogen.

4. Better impact results were obtained on the above parts when transformation of austenite was incomplete than when it had been completed.

5. The brittleness encountered in cyaniding S.A.E. 6140 was not due to carbon penetration, since carburized parts gave extremely high impact results.

6. The poor properties imparted by oil quenching S.A.E. 52100 from cyanide, and then drawing, were improved by using an interrupted quench in hot oil.

7. Strangely enough, a straight oil quench and draw on S.A.E. 6140 which had been heated in cyanide gave better results than austempering from cyanide. However, samples heated in a controlled atmosphere furnace showed greater toughness upon austempering than after an oil quench and draw.

8. Although S.A.E. 6140 is supposed to give its lowest impact properties in the range between 500 and 700° F., quenched samples drawn in this range gave results which were almost as good as those exhibited when drawing at 400° F.

9. Impact results on the Izod bars listed under the treatments of series No. 5 showed two things: *(a)* Bars given a cyanide case were inferior in toughness to bars heated in a controlled atmosphere; *(b)* cyanided bars which were ground to size and notched after heat treatment proved inferior to identical bars heated in a neutral atmosphere. This series of tests showed that

heating in cyanide affected the core properties of the steel.

10. We were unable to find any suitable explanation for the embrittling action of a cyanide heat. Nitrogen determinations run on samples heated in cyanide and in a controlled atmosphere furnace were identical, proving that nitrogen does not penetrate into the core during a cyanide heat.

11. Neither the case nor core structures of samples of S.A.E. 6140, heated in cyanide, showed any unusual appearance, being normal in all respects. The core structure showed evidence of undissolved carbides, but since more rapid heating is obtained in cyanide than in a controlled atmosphere furnace, one cannot base any conclusions on that point.

Conclusion — It has been the intention of this report to present data and information on the impact properties of steels heated in cyanide. During the course of the investigation we ran various other treatments in an effort to achieve optimum results. No pretense is made to explain the reasons for the poor performance of cyanide treated parts under impact. Where wear resistance is the sole requirement, a cyanide heat is of great value, but if the impact is also present, one should proceed with caution. Although cyanide seems to embrittle the parts made from S.A.E. 6140, we have continued to use it as a heating medium because of the lack of a substitute which will equal it in the wear resistance at high temperatures which is so essential on this particular part.

Aircraft

S. A. E. Meeting Discusses Prestressing, Sleeve Bearings and Magnesium Sheets

By Our Special Correspondent

A METALLURGICAL observer at the Society of Automotive Engineers' "National Aeronautic Meeting" (New York City, April 3 to 5) found at least three topics of active interest to the readers of *Metal Progress* — prestressing of metal parts to improve their performance in service, tests on heavy duty sleeve bearings for engines, and the use of magnesium for structural members.

As to the first subject, an especially interesting paper was contributed by R. G. Anderson of the Cleveland Development Division of the Aluminum Co. of America, "Improving Engine Parts by Direct Measurement of Strain". It contained a stimulating discussion of the value of stress analysis as an aid in increasing the life and useful strength of machine parts. The great importance of knowing and controlling residual stresses was pointed out, because if the residual stresses have the same direction as the imposed operating stress, the useful strength of the member will be reduced by the amount of the residual stress; if, on the other hand, the residual stress is opposed to the working stress, the strength of the member will be increased a corresponding amount.

Mr. Anderson classified the principal stresses in an operating machine and their origins as follows:

1. Imposed stresses from operating loads.
2. Residual stresses in the part retained from nonuniform plastic deformation caused by *(a)* quenching, *(b)* machining, and *(c)* nonuniform cold plastic yielding during fabrication, assembly, and operation.
3. Prestresses introduced during assembly.
4. Thermal stresses set up during operation as a result of heat gradients.

The principles involved were illustrated by several comprehensive examples, one of which was a complete discussion of actual test results showing the benefits to be derived from the prestressing of bolts. It was also shown how a rotating disk can be stressed through overspeeding severe enough to cause plastic flow of the metal surrounding the central hole. This plastic flow is

due to tensile stresses from the centrifugal force; the stresses increase from the periphery toward the center and localized plastic flow begins as soon as the stresses exceed the yield point. When the disk is stopped and the centrifugal stress dies down to zero the material surrounding the center hole will retain compressive stresses whose magnitude and distribution depend on the extent of overspeed and overstress. These compressive stresses will reduce the tension stresses resulting from subsequent rotation of the disk at speeds below the prestressing overspeed.

Also described was an ingenious method of strengthening a flanged tube joint by dishing the mating surfaces. This places a compressive prestress in the area where the tube fairs into the flange. Upon application of an axial load no tension stress can arise in the radius between the tube and the flange until this compressive prestress has been balanced by the tensile load.

Prestressing is not the only method of increasing the stresses that can safely be imposed upon the material. Improved design can accomplish a decrease of harmful prestress — for instance, when the latter is due to temperature gradients. This method was illustrated by data on temperature distribution and thermal stress in cylinder heads of two different designs.

Mr. Anderson noted that the experimental methods employed by stress analysts may be either qualitative or quantitative. Brittle lacquers are used for the preliminary qualitative stress analysis to locate highly stressed areas which later on are covered by strain gages to obtain quantitative measurements of the principal stresses. Prestress can be determined by drilling one or several holes in the material with a sharp drill and accurately measuring their changed size and shape, or by mounting strain gages in the desired location and progressively removing metal from the assembly whose internal stress is to be measured.

He pointed out, finally, that many failures of machine parts in service are due to unfavorable prestress rather than to defective material. In general, machine parts contain a sufficient amount of material although often the material is inefficiently distributed. Many inexperienced diagnosticians, who might be too prone to attribute material failures to minor imperfections in the microstructure, would do well to study the reasoning and methods presented in this paper.

Sleeve Bearings

J. Palsulich and R. W. Blair of Wright Aeronautical Corp. presented a paper dealing with the impressive and complicated equipment employed in the development of highly loaded sleeve bearings and bearing materials for Wright engines. Properties investigated are:

1. Maximum load-carrying capacity.
2. Friction and lubrication characteristics.
3. Fatigue life.
4. Corrosion resistance.
5. Wear resistance.
6. Cavitation erosion resistance.

Unfortunately, this paper had scant information about the composition of the several bearing materials which have been tested and whose various properties have been evaluated — possibly because the authors may have believed that composition was of less importance than manufacture, installation and maintenance. It was stated that the test data were in good agreement with subsequent tests on a full scale engine, but a study of the detailed test results listed opposite the materials did not satisfy the outsider's curiosity as to how the test results could be correlated and inter-

300-Mi. per Hr. Lockheed "Constellations" for Chicago to England Route. 60 passengers, 10,000 hp., 123-ft. wing span, 25,000-ft. ceiling, controlled atmosphere in cabin, 50 tons gross at take-off

preted. The lasting impression given by this paper was focused on the ingenuity and attention to detail which characterized the various test procedures and testing machines rather than on factual information regarding the properties of different bearing materials.

Magnesium

Two papers devoted to the lightest of structural metals led to lively discussion pro and con. J. E. DeHaven of Battelle Memorial Institute spoke on the "Development of the Magnesium Alloys as Aircraft Materials", and gave an interesting outline of the history and background of the material as well as a general account of recent improvements and progress in alloying. He considered the new high purity alloys to be vastly superior in corrosion resistance to the older less pure materials. Iron turns out to be especially harmful but copper, nickel, and silicon also are undesirable although less frequently encountered. The corrosion resistance of such pure magnesium alloys to salt atmosphere has been greatly improved. New alloys such as the one containing 6% cerium and 2% manganese have surprisingly good mechanical strength properties at temperatures up to about 600° F. Especially in the higher temperature ranges, they are graded by Mr. DeHaven as superior to any known aluminum alloys. By alloying magnesium with a small amount of beryllium, the flammability of the metal has been reduced to such an extent that such a magnesium sheet subjected to incendiary ignition was affected little more than steel. Although the future of the metal seemed to hold great promise, the author did not overlook mentioning the many pitfalls, detours, and blind ends encountered by the investigator who is attempting to develop an alloy meeting the strength and fabrication requirements of present-day aircraft constructors.

J. C. Mathes of Dow Chemical Co. (Earl Rottrayer of University of Michigan, co-author) presented the concluding paper on magnesium. It was entitled "A Summary of Design and Test Experience With Monocoque Magnesium Wings". This paper was well illustrated with views showing the design details and methods of test loading of monocoque wings for the PV-1 airplane. (For the benefit of metallurgists not acquainted with aeronautic jargon, the monocoque structure is one which carries the structural stress in the skin of the member rather than in internal braces and stiffeners.) This paper revealed the interesting fact that the magnesium wing failed at 113% of the ultimate load and 170% of the limiting load (maximum experienced by the member in flight), whereas the aluminum wing of conventional design failed at 90% of the ultimate load. Since the weight of the magnesium wing was 700 lb. and the comparable aluminum wing 650 lb., the 8% greater weight resulted in 25% greater load-carrying capacity. The original objective was not completely realized since early experiments indicated the necessity of incorporating some internal bracing into the monocoque in the form of spars and fanwise stiffeners to eliminate buckling. The project, however, did show that the development of magnesium alloys has progressed to the extent where they may soon be in actual competition with aluminum-base metals in fields previously dominated by the latter.

These two papers were discussed simultaneously and aroused considerable comment. Representatives of the airlines voiced a conservative attitude with regard to the increased use of magnesium sheets in commercial aircraft structures. (Castings, forgings, and extrusions are more acceptable.) Instances were cited where the low impact strength of magnesium alloys had resulted in numerous failures of secondary equipment such as furniture. Corrosion resistance was also considered unsatisfactory for exterior applications such as door panels. From the airlines' side, it was also stated that manufacturers are quite prone to quote service experience gained on military planes, forgetting that even 1000-hr. flight time was unusual on military equipment but the airlines expect a service life ranging between 15,000 and 20,000 hr. Some misgivings, finally, were expressed with respect to the flammability of magnesium sheets.

From the side of airplane manufacturers, it was pointed out that magnesium wheels are used on all large transports and have been most satisfactory. Other magnesium parts of long standing are engine castings, cylinder heads, and about 400 to 500 different castings used on housings and accessories. Past failures frequently have been due to ignorance of the differences between magnesium and the metals for which it was being substituted. Not only does magnesium require design modifications, but it likewise requires modified manufacturing methods. For instance, operators accustomed to riveting aluminum require retraining before they are competent to rivet magnesium sheets. It was also mentioned that the cycle weld that had been used to attach soundproofing material to cargo door panels of magnesium had been known to induce corrosion because of the release of free halogen ions from the cement upon exposure to moisture. Magnesium is not suitable for cargo compartment liners because of its low hardness and low impact strength.

Bronze

A New Copper-Phosphorus-Lead-Nickel Alloy

By Vladimir A. Grodsky
Metallurgical Engineer, U. S. Naval Gun Factory, Washington

PHOSPHOR BRONZES belong to one of the most commonly used classes of bronzes and their melting technique and properties are well known. The name is somewhat misleading because these alloys contain only small amounts of phosphorus. For instance, gun metal, hydraulic bronze, and valve bronzes are permitted 0.05% P max., some bearing bronzes 0.6% P max., gear bronze (standard bronze) 0.25% P max., and phosphor bronze 0.5% P max.

Of all the nonmetallic elements used for deoxidizing metals, phosphorus is the most active and therefore a phosphorus-copper hardener makes a very powerful and efficient reagent and is commonly used for producing phosphor bronzes. These phosphorus-copper hardeners ordinarily contain 10 and 15% phosphorus, and have melting points of approximately 1560 and 1870° F., respectively. They have a coarse crystalline structure, are very hard and brittle, have a dull coppery color and a gray fracture. It is advisable to acquire the hardeners ready made, because of their difficult manufacture.

Looking at the copper-phosphorus constitutional diagram (Fig. 1) worked out early in this century by Heyn and Bauer, it can be seen that saturation is reached at 1870° F. (1022° C.) and a phosphorus content of 14.1%. This analysis corresponds to the formula Cu_3P. There are a few other compounds of phosphorus and copper but the most interesting and the most important is Cu_3P with a melting point of 1870° F. and the hardness of hardened toolsteel.

All the high phosphorus-copper alloys are brittle and hard, and the diagram shows that only in the low-phosphorus end of it could one expect to take advantage of the physical properties of a copper-phosphorus alloy. When phosphorus exceeds 1.7% a eutectic appears in the alloy, as can be seen from the diagrams. This eutectic consists of the maximum solid solution of phosphorus in copper plus the phosphide Cu_3P. Therefore, copper alloys with phosphorus between 1.7 and 14% represent a eutectic system. However, an excess of phosphorus over 6% makes the alloy very brittle with decreased tensile strength and low elongation.

When a copper-phosphorus alloy begins to freeze, a dendritic crystallization of the solid solution starts to form from the outside surfaces inward. In accordance with well-known facts the space between the dendrites is filled with smaller amounts of copper-phosphorus compounds having a lower melting point. This increases the density of the casting to a certain extent. In order to raise the density still higher some plastic substance, which has a low melting point and does not form any compounds or solid solutions with the basic components, has to be used. Such a substance is lead, and its use eliminates the need for tin for the same purpose.

The metal tin has been

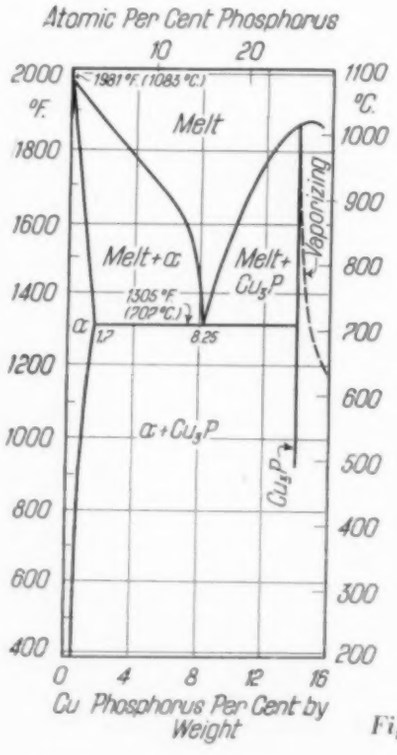


Fig. 1 — Copper-Phosphorus Diagram

very scarce since the beginning of the war and still is; furthermore, it has a lower melting point (450° F.) as compared with 621° F. for lead. Its use would lower the final solidification temperature of the alloy considerably and increase the possibilities of segregation. This would diminish the usefulness of a tin-bearing alloy for many classes of work.

The copper-lead diagram (Fig. 2) shows a eutectic point at 619° F. (326° C.) where the separation of copper stops and final solidification takes place, the remaining eutectic containing only 0.06% Cu. The liquidus after the reaction at

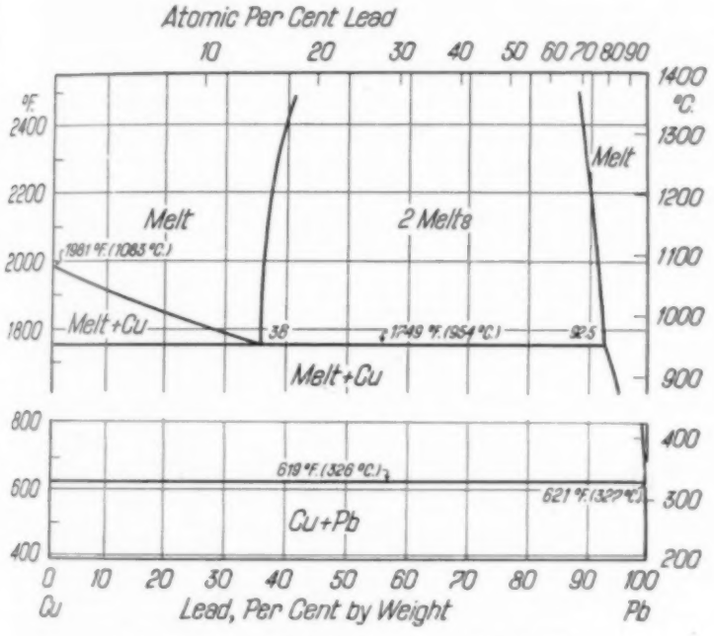


Fig. 2 — Constitution of Copper-Lead Alloys

1749° F. (954° C.) contains about 7.5% Cu; copper therefore crystallizes from the melt while the temperature falls from 1749 to 619° F.

It can be seen from both diagrams, copper-phosphorus and copper-lead, that when lead is added to the copper-phosphorus alloy, the solid solution of phosphorus in copper will form a eutectic with lead as well as with copper phosphide. There exists also the possibility of forming a third eutectic, which besides lead and Cu_3P will contain α solid solution. Lead distributed in the eutectic will, to a certain extent, compensate for the brittleness of the copper phosphides, but as lead increases, the strength of the alloy diminishes. However, the lead content is not as important as its distribution. The smaller the lead globules are and the more uniformly they are scattered throughout the mass of the alloy, the greater will be the density, and this property makes the alloy especially suitable for pressure castings.

It is also known that nickel forms a continuous series of solid solutions with copper, free from phase changes. Nickel also greatly increases the dispersion of lead in copper-lead alloys, and therefore a little nickel is added to the new alloy.

Following the above lines of reasoning a large number of experimental melts have been made in the U. S. Naval Gun Factory, their chemical composition being within the following limits:

Phosphorus	1.0 to 10.0%
Lead	5.0 to 30.0
Nickel	0.5 to 3.0
Copper	Balance

It has been found that for ordinary castings the best results were obtained from alloys containing from 2 to 4% P, from 8 to 12% Pb and from 0.75 to 1.5% Ni. As is to be expected, alloys with more than 4% phosphorus are brittle, although their hardness increases considerably as the phosphorus goes up.

The tabulated figures below show that the Cu-P-Pb-Ni alloys are well suited for castings whose physical requirements need not be very high. However, these alloys also have other properties such as density, machinability, weldability and corrosion resistance which are often more important than strength.

Foundry Practice

The melting procedure is as follows: Copper and nickel with about half of the amount of P-Cu hardener are melted in a crucible. When the metal is all melted (700 lb. in about 2 hr.), the rest of the P-Cu hardener is added and the melt heated to 1850 or 1900° F. After this temperature is reached, the melt is cooled to about 1800° F., the required amount of lead is added and the melt

Typical Properties of Copper-Phosphorus-Lead-Nickel Alloys

Phosphorus	10.0	7.0	6.5	6.0	6.0	5.5	4.0	3.25	2.0
Lead	10.0	12.0	7.0	15.0	10.0	5.6	9.6	10.0	8.0
Nickel	0.5	1.0	1.5	1.0	1.2	1.8	2.2	1.0	0.5
Copper	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance	Balance
Tensile strength	23,000	59,000	22,300	38,900	40,000	49,500	47,600	43,200	30,000
Elongation	2.5	0	2.25	0.4	1.0	1.5	7.9	13.25	19.0
Rockwell	B-94	B-90	B-71	B-79	B-77	B-71	B-54	B-46	B-40

vigorously mixed. The metal will then have a temperature of about 1750° F. and is ready to be poured. It is advisable to throw in a handful of P-Cu shot just before pouring.

Best results are obtained when the alloy is melted, cast into pigs, and then remelted to pour castings.

The alloy has a melting temperature of about 1400° F., is very fluid and searching, and should be poured always at the lowest possible temperature — 1700 to 1750° F.

No flux or charcoal cover is required while melting.

The metal can be cast in cast iron or sand molds. Dry sand and green sand molds can be used, but the latter must be skin dried. No special requirements for the sand are necessary. Segregation will occur when the pouring temperature is too high or the moisture content of the sand is excessive. Chills can be used and the mold does not require many heads and risers on account of the fast freezing characteristics of the new alloy.

The alloy can be easily welded, by gas or electricity, with rods made from the same metal. Bare welding rods produce a clean, homogeneous electrical weld, without the use of a flux. For best results the phosphorus content of filler rod or electrode should not be below 6%.

Summary

The developed alloy, which will doubtless be further improved, has many advantages:

1. It has a low melting temperature — about 1400° F. min., which cuts down the melting time about one-third as compared with phosphor bronze. No flux is required during melting.

2. The casting temperature is low: 1700 to 1750° F.

3. The metal is very fluid, fills out the mold very well and has a low shrinkage.

4. Dry sand — or skin dried, green sand molds — can be used, as well as chill molds. Fewer heads and risers are required.

5. The alloy has a high density and is very well suited for pressure castings.

6. Its machinability is very good.

7. Physical properties are

Tensile strength	30,000 psi., min.
Elongation	6% min.
Brinell hardness	45 min.

8. Corrosion resistance of the alloy is higher than that of phosphor bronze in ordinary exposures in service.

9. The alloy can be easily welded with gas or electricity. Welding rods without any flux or coating produce homogeneous welds.

10. The bearing properties can be favorably compared with the performance of the best copper-lead-tin bronzes.

11. No tin is used in the making of the alloy — although small amounts of tin and zinc are not harmful, making it possible to remelt scrap containing a little of these elements.

12. The cost of the alloy is low on account of the absence of tin, shorter melting time, a simpler molding technique, and the absence of heat treatment.

The alloy has been developed at the U. S. Naval Gun Factory's brass foundry with the advice and help of J. E. Crown, master mechanic, P. O. Ady, quarterman, and the foundry supervisors and men. The Navy Department, Bureau of Ordnance, has covered the alloy by Specification 1659, approved May 18th, 1945.

Shallow Draft Rowboat, Hull Stamped From Single Sheet of 0.064-Gage Aluminum Alloy, Weighs 150 Lb. Without Outboard Motor. Floats 6 Men or 1200 Lb. Courtesy Reynolds Boat Co.

Influence of the "Modifier" on the Nature of Aluminum Alloys

Moscow, U.S.S.R.

To the Readers of METAL PROGRESS:

In my letter published in the January issue I have described some of the results of work done in our laboratory by Messrs. Sergeiev and Polack in a study of the viscosity of molten aluminum and its alloys. Now I can report on the effect of the alloying element sodium on the viscosity of Al-Si alloys, especially the eutectic Al-Si alloy "modified" with sodium known as "silumin".

Modification was carried out by introducing small quantities of metallic sodium (0.05, 0.1 and 0.2%) or a mixture of its salts (⅔ NaF with ⅓ NaCl) amounting to 3% of the alloy while the melt was at 760 to 800° C. (1435° F.).

The introduction of the modifier was always followed by a very large increase of the viscosity. During the first moment after modification the ball would not oscillate. (Refer to the diagram of the apparatus given in my former letter.) Five to seven minutes later the measured coefficient of viscosity was still 15 times as high as that for the eutectic Al-Si alloy in the initial state.

A decrease in the viscosity of modified silumin corresponds in time with the decrease of the modification effect observed in foundry practice when a modified alloy is soaked or held in molten condition for too long a time. The considerable increase of viscosity observed upon modification far exceeds the range of viscosity of the binary alloys as they depend on temperature and composition [plotted in the diagram on page 99 of the January issue] and is certainly the result of internal changes in the liquid state of the alloy produced by sodium. See Fig. 1, herewith.

This effect was also observed in commercial aluminum (99.5% Al). Its viscosity coefficient measured 5 min. after the introduction of 0.1% sodium (no change in temperature) is 0.627 units —approximately 17 times as high as the value 0.037 given for aluminum in my former communication.

There is a similar effect on the viscosity of aluminum produced by magnesium and calcium, as is shown in Fig. 2. By soaking of alloys after the addition of these elements the viscosity decreases, but more slowly according to the slower rate of their removal by evaporation or oxidation.

Many other elements, such as tin or titanium, produce no effect on the viscosity of aluminum. (See Fig. 2.) Measurements of the surface tension of the alloy show that this violent effect on viscosity is a characteristic for elements which may be said to be "surface-active".

The modification process gives not only such positive results as grain refinement, with its corresponding increase of mechanical characteristics of the alloy, but also negative ones such as increase of porosity of the castings. The latter is viewed by many authors as a result of an increase in gas content brought in, dissolved in the sodium added. However, in our laboratory Messrs. Sergeiev and Chomchenovskaya have modified silumin by add-

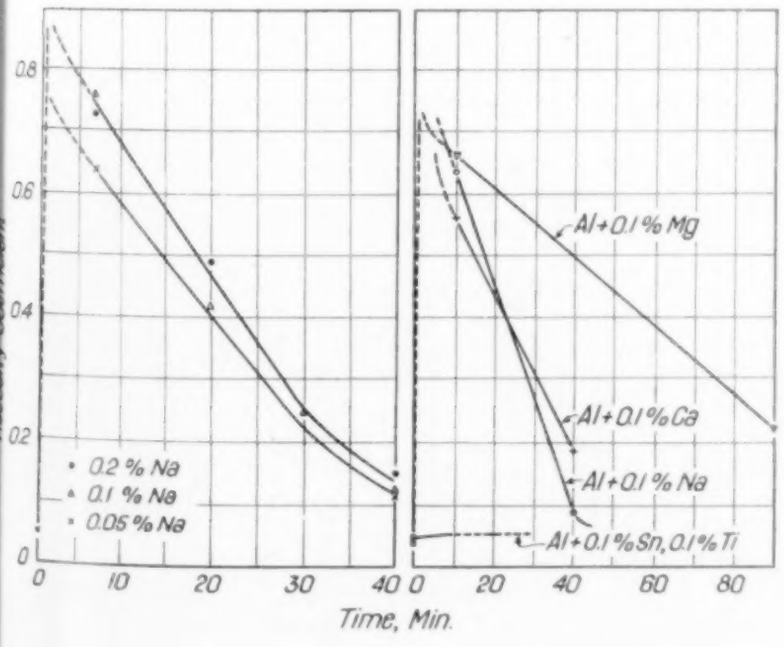


Fig. 1 (Left) — Variation of Viscosity of Modified Al-Si Eutectic Alloy With Time After the Addition. Fig. 2 (Right) — Variation in Viscosity of Pure Aluminum With Time After the Addition of Small Amounts of Sodium, Calcium or Magnesium

ing sodium which had previously been repeatedly distilled in a high vacuum and thus thoroughly degasified. Nevertheless, even in such tests, samples cast in dry molds under conditions preventing any absorption of gas, were more porous than unmodified ones.

The data obtained on the viscosity of modified silumin and the experiments described above show that the causes of the porosity increase of castings upon modification lie not only in the increased degree of gas saturation of the alloy but also in the nature of the gas evolution from the alloy. When viscosity increases 15 times, the gas evolution from molten alloy would be 15 times slower. Consequently the substantial increase of viscosity of modified alloy is one of the direct causes increasing the porosity of cast silumin.

The data obtained are also interesting in the light of the study of the mechanism of the modification process.

The marked increase of the alloy viscosity observed upon modification calls to our mind the possibility of the existence of some relation between the modification effect and the structural changes originating from the liquid state. It is the author's belief that this assumption is to be considered with other existing hypotheses when studying further the nature of the action of a modifier.

G. Akimow
Physical Metallurgy Laboratory
Research Institute for Aircraft Materials

A Workable Classification of Toolsteels

Dayton, Ohio

To the Readers of Metal Progress:

The undersigned has read with much interest the article by C. L. Hibert in the February issue ("A Workable Classification of Toolsteels") and has discussed it with a number of the members of the metallurgical organization of General Motors Corp. Hearty accord was expressed with the author's approach to the problem; however, it is deemed wise to point out a few issues that may cause its readers trouble and confusion.

At the outset, it is generally accepted by contemporary metallurgists that chemistry alone will not constitute a satisfactory specification for toolsteels. It is believed by metallurgists in General Motors' organization that hardenability, porosity, grain size, and carbide distribution are the important factors governing their quality. Consequently, G.M. standards have been built around this premise. Furthermore, we do not consider S.A.E. 1020 and 1050 as "toolsteels", whereas Mr. Hibert lists them as such in his Table I under code of Circle 1 and 2. Circle X ("Graph-Mo") steel should certainly be listed in Group 2, since it is definitely an oil hardening, nondeforming toolsteel.

In April 1945 a Joint Industry Committee consisting of representatives from all large automotive corporations, tool manufacturers, toolsteel manufacturers, and other large heavy industries, agreed upon a standard tungsten-molybdenum high speed steel that could be manufactured and marketed nationally. Its analysis is as follows:

Carbon	0.79 to 0.86%
Chromium	3.90 to 4.40
Molybdenum	4.75 to 5.25
Tungsten	6.00 to 6.75
Vanadium	1.75 to 2.05

This analysis has been in production in the mills since that time, and it is unlikely that Mr. Hibert's Triangle 3 would now be obtainable. It may also be pointed out that Triangle 3 would be inefficient with a carbon content less than 0.78%. However, it is the hope of the Joint Industry Committee that *all* consumers of standard high speed steels will eventually turn to the above M-2 type.

We must also challenge the published results on impact (comparison curves shown for Triangle 1 and Triangle 3, nationally known as T-1 and M-2 respectively). It is not known whether notched or unnotched specimens were tested, but unnotched specimens of M-2, hardened at the correct temperature of 2225° F., have an average of 135 in-lb. impact compared to 100 in-lb. for T-1 hardened at 2325° F. Both steels were double tempered at 1050° F. Converted to ft-lb. this would be 11.3 and 8.3 respectively compared to the author's comparison of 12 to 18 respectively. The former figures have been duplicated by Crucible Steel Co. of America in a tremendous amount of testing.

For oil hardening toolsteel Mr. Hibert recommends quenching to 200 to 300° F. with an immediate temper. At 300° F. this type of steel is only 80% transformed to martensite. Cracking may actually occur after the temper. It is rather to be recommended that this material, like all other steels, be quenched to 150° F. or thereabouts.

These criticisms probably reflect the attitude of an organization that engages in far more machining activity than Mr. Hibert's (aircraft). He is to be congratulated on a splendid and informative piece of work; undoubtedly his classification saved his staff much grief and his company much money. This letter is written in the hope that misunderstanding and trouble may be avoided in the instances cited.

Stewart M. DePoy
Chairman, General Motors' Toolsteel Committee
Delco Products Division

Raised Yield Strength in Bend Tests

LAGUNA BEACH, CAL.

To the Readers of METAL PROGRESS:

In the course of a continuing study of the phenomena reported in last December's issue ("Raised Yield Strength in Bend Tests") it became apparent that the displaced location of the stress-strain curve in bending as compared to the position of the stress-strain curve in tension could be predicted mathematically by methods analogous to those used in my study of residual stresses in wire loops at anchorage shoes or grommets (*Metal Progress*, September 1943). Approaching it from this angle it developed that the tangent point in the stress-strain curve in bending is apparently raised, but it also became apparent that a 1.2% increase in testing machine load accounted for the 23% increase in proportionality measured.

It therefore may be concluded that the tests described in the December article are not accurate enough — indeed it would need much more precise equipment than is now available — to prove unequivocally that the *tangent* yield is actually raised in any bend test. However, that does not affect the experimental data presented in the paper nor indicate that the practical conclusion is in error. The latter is that the limiting design values used with the beam formula in bending can be raised substantially above those values in tension. This conclusion should be re-emphasized because the larger design values have been so used with complete success on two important pieces of equipment that I know of.

GIVEN BREWER
Consulting Engineer

Metal-Wood Combination

STRATFORD, CONN.

To the Readers of METAL PROGRESS:

A metal-wood-metal sandwich material that has proved its worth in aircraft construction may well have many applications in other industries requiring light rigid construction. This is known as "Metalite"; it was developed by Chance Vought engineers and has been used for stabilizers for the F4U-4 Corsair in production quantities. It consists of two thin sheets of any strong aluminum alloy as required by the load and exposure demanded of the part, separated by a thin core of balsa wood blocks set with grain perpendicular to the sheet. All parts are held together by a strong thermosetting adhesive. Flat sheets (or slightly curved shapes) are assembled and held to form in appropriate clamps and bonded in an autoclave operating at moderate heat and pressure.

Stiffness, smoothness, lack of skin wrinkles, and freedom from joints and fasteners that interfere with uninterrupted airflow — these characteristics also may well be of great advantage to various other mechanical or structural designs.

F. J. LELEAR
Chance Vought Aircraft Division
United Aircraft Corp.

Nature of the Anodic Oxidation Film

PARIS, FRANCE

To the Readers of METAL PROGRESS:

Anodic oxidation is the best method of surface protection for aluminum and its light alloys. In practice, certain defects occur on the surface — like a mesh or spider-web in nature. The cause is unknown. In order to determine their origin and thus to find a remedy, the first step is to determine the nature of the protective film resulting from anodic oxidation.

Such an X-ray and optical investigation has been carried out by Messrs. Lacombe, Beaujard and Morize. They examined both samples of the anodized metal and samples of the film alone, isolated from its metallic base by anodic solution of the latter in a bath of acetic anhydride and perchloric acid used for electrolytic polishing. The latter method permits an extension of the electron microscope technique of examining very thin films (imprints of the surface condition) to thicker and less fragile oxide films. The researchers concluded that this aluminum film is an aggregate of microcrystals subject to a double orientation effect:

1. A common electrolytic effect with the axis of the "fiber" perpendicular to the oxidized surface -- in other words, following the equi-potential lines as observed in electroplated metal coatings.
2. A crystalline orientation of the grains of the metal, also as observed in electroplates.

This leads to an explanation of the surface appearance of metals protected by anodic oxidation, and at the same time sheds some light on the origin of the defects in question.

"Mesh" may be described as a marbleized structure of alternating gray and white on anodically oxidized surfaces of aluminum-magnesium alloys, such as shown in Fig. 2, photographed double size. It is caused by irregularity or heterogeneity of the precipitation of Al_3Mg_2 at the *beginning* of tempering at *low* temperature, this compound being blackened by anodic oxidation. This irregularity of precipitation is shown microscopically in Fig. 3, page 976, and it derives from the uneven concentration of the mother solid solution originating in the dendritic segregation during solidification; it therefore is macrographic

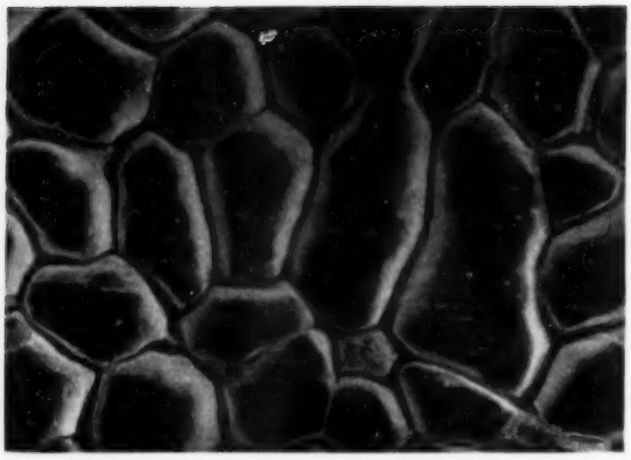


Fig. 1 — Appearance of the Oxide Film on a 9% Mg-Al Alloy, Tempered 3 Hr. at 400°F. Black light, 350×. (Lacombe and Beaujard)

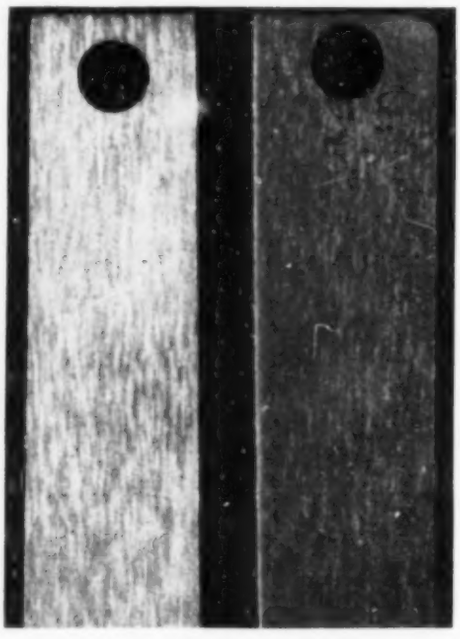


Fig. 2 — Appearance of Mesh, Twice Size, on 9% Mg Alloy, Tempered 2 Hr. and 5 Hr. at 460°F. (Lacombe and Morize)

in character. As the time of tempering is prolonged, the mesh disappears, since the precipitation extends over the entire surface of the grains, thus giving a uniform color to the oxide film.

"Spider web" structure is a network of fine lines or furrows and hollows visible either as black lines on a light background, or as white lines on the metallic background, and forming cells more or less clearly oriented, generally in the direction of working. Herenguel has concluded that this defect originates in a moderate mechanical deformation of the as-cast grains or grains of primary solidification, which causes intercrystalline cracking (as well as transcrystalline slip) that is concealed by the surface work [flattening with lapping] but which is revealed by the anodic attack.

To summarize, *mesh* is related to the primary solidification structure and to heat treatment; *web* is related to primary solidification structure and to mechanical treatment.

In both cases there exists a hereditary influence of the solidification structure or as-cast macrostructure which is revealed by anodic oxidation acting as a macrographic etching reagent, on condition that there has occurred a previous sensitization by an appropriate thermal or mechanical treatment. (It might be noted in this connection that in order to show the lines of deformation in mild steel by means of copper reagents a preliminary sensitization is necessary by tempering at low temperature to produce a heterogeneous precipitate.)

Thus not only is anodic oxidation shown to be a new macrographic technique for light alloys, but it is also actually the most sensitive method for disclosing the first traces of precipitation and of heterogeneity in the aluminum-magnesium solid solution at the beginning of tempering or aging after quenching. It has been shown that when the ordinary microscopic and X-ray methods of examination give no indications of any change, anodic oxidation will attack, preferentially, the solid solution's crystal boundaries, since Al_3Mg_2 has begun to precipitate there — on condition that anodic oxidation has been preceded by electrolytic polishing.

Anodic oxidation of the light alloys, under the conditions outlined above, therefore becomes both a macrographic reagent for revealing defects in the alloys and a particularly sensitive micrographic reagent.

ALBERT PORTEVIN
Consulting Engineer

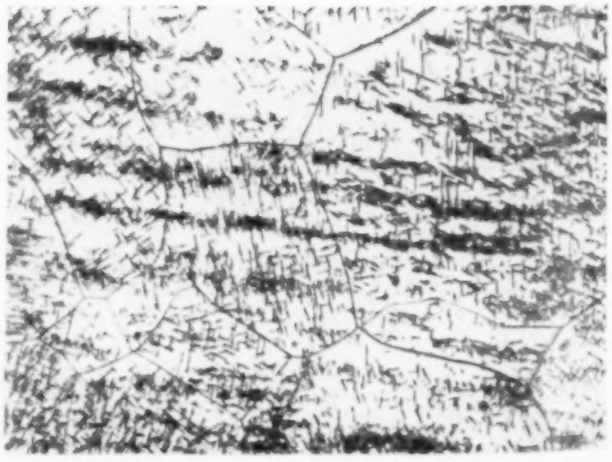
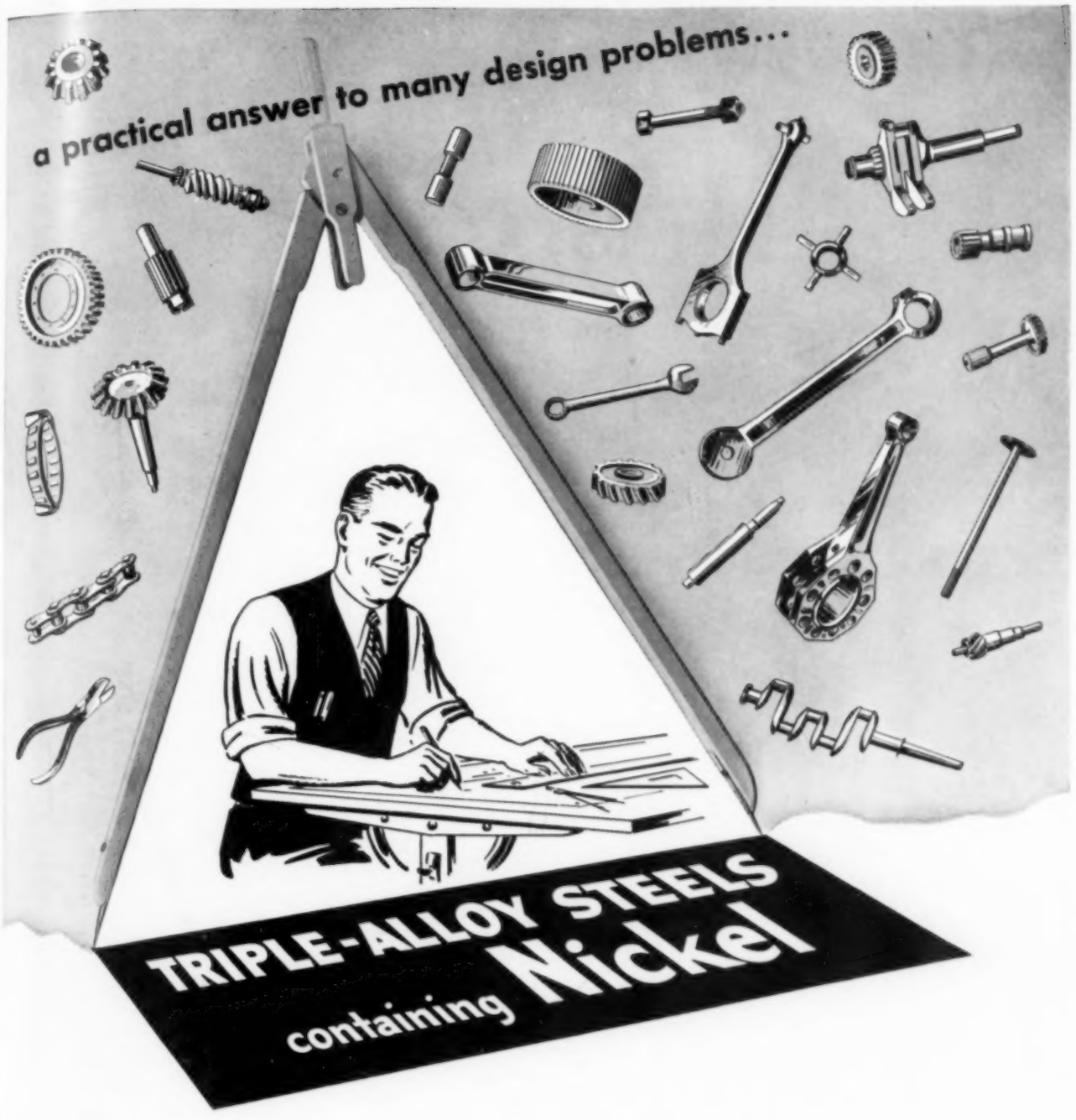


Fig. 3 — Heterogeneity of Precipitation of Al_3Mg_2 in the 9% Mg Alloy, Tempered 2 Hr. at 460°F. 500×. (Lacombe and Morize)



Triple-alloy steels containing Nickel offer designers the following *triple* advantages:

1. OUTSTANDING PERFORMANCE—Strength and toughness, resistance to wear, fatigue or shock to meet a wide range of requirements, as dictated by design.
2. RELIABILITY—based on consistently uniform response to heat treatment.
3. ECONOMY—resulting from standard compositions precisely graded to match the engineers' needs.

Service records established by triple-alloy steels over a period of years show that they are giving excellent results in many diverse and exacting applications.

The large number of standard compositions available, including the 4300, 8600, 8700, 9300 and 9800 series, permit accurate and economic selection for specific uses.

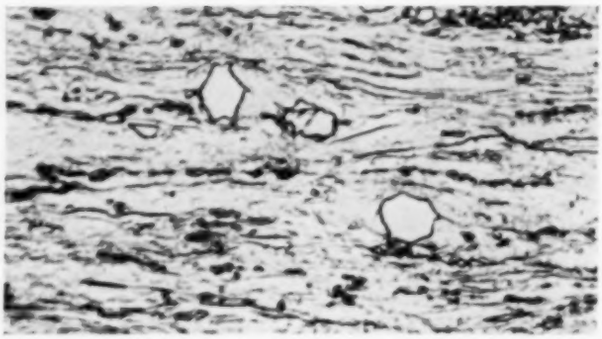
Because of their many advantages, these triple-alloy steels warrant your careful consideration when planning new or improved designs. We shall be glad to furnish counsel and data upon request.

THE INTERNATIONAL NICKEL COMPANY, INC. 67 Wall Street New York 5, N.Y.

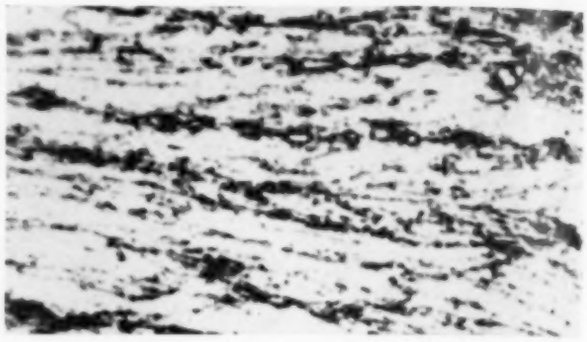
Development of Sigma Phase in 27% Cr-Fe

By James J. Heger

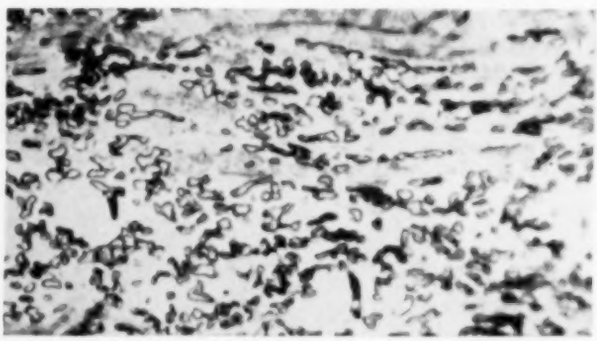
Research Dept., The Babcock & Wilcox Tube Co., Beaver Falls, Pa.



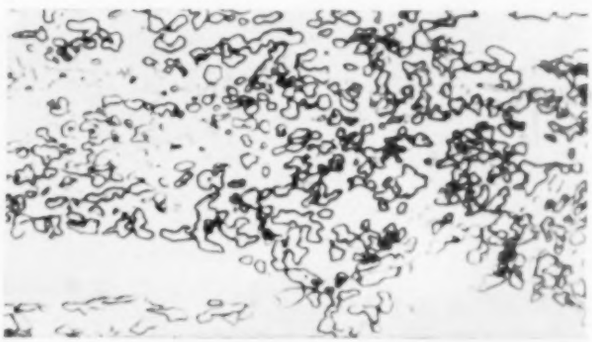
Ferrite Flowed Around Hard Carbide Particles After Cold Working 80%; Hardness C-24. 1000 ×



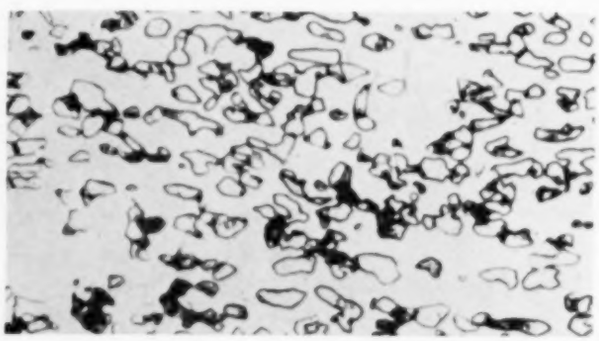
Very Fine Precipitate After Aging 1000 Hr. at 885° F.; Hardness C-39. 2000 ×



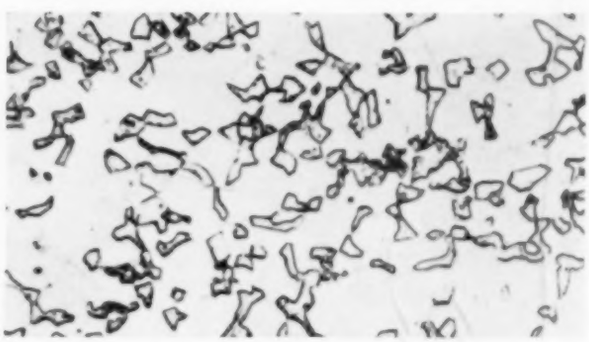
Precipitated Particles Coalescing After Aging 1000 Hr. at 950° F.; Hardness C-37. 2000 ×



Substantial Coalescence of Sigma Phase After Heating 1000 Hr. at 1000° F. 1000 ×



Hardness Is C-34 After 1000 Hr. at 1050° F. 2000 ×

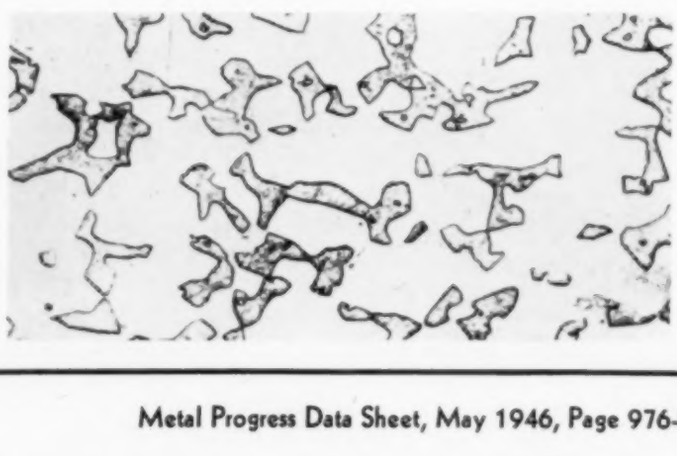


Aged 1000 Hr. at 1100° F. 1000 ×

MATERIAL:
½-in. tube,
4½ o.d.

ANALYSIS:

C	0.13%
Mn	0.48
Si	0.47
Cr	26.95
Ni	0.17
N_2	0.16



Aged 1000 Hr. at 1200° F. 2000 ×

All sections lightly etched (1 to 2 sec.) with aqua regia

Properties and Characteristics of 27% Chromium-Iron

By H. D. Newell
Chief Metallurgist, The Babcock & Wilcox Tube Co., Beaver Falls, Pa.

THIS PAPER represents a compendium on the mechanical properties and structural characteristics of 27% chromium-iron alloy. The studies outlined herein were made originally for Rubber Reserve Co., now "Reconstruction Finance Corp., Office of Rubber Reserve", and were intended primarily for manufacturers of raw materials for synthetic rubber. The relative chemical inertness of the 27% alloy under alternate reducing and oxidizing conditions, such as are encountered in the catalytic dehydrogenation of N-butane, has warranted the use of this material for equipment for making butadiene from petroleum.

Much of the experimental data cited were derived from research work conducted under contract by The Babcock & Wilcox Tube Co. for Rubber Reserve Co.* These data not heretofore available are supplemented by information from other sources, as acknowledged throughout the text. Included also is a summary of the metallographical examinations on materials following service in dehydrogenation operations. Detailed information on weldability has been omitted, due to lack of space. Unless specifically stated otherwise, all considerations pertain to the wrought forms of 27% chromium-iron containing about 0.25% maximum carbon.

Description of the Alloy

A.I.S.I. Type 446 stainless steel is generally classified as a heat resistant alloy. Owing to its high chromium content, however, it may be regarded also as a corrosion resistant alloy. The broad range of composition currently specified by the A.I.S.I. and A.S.T.M. for tubular and flat products of Type 446 steel is given in Table I, page 978.

Usually, 27% chromium-iron tubing is furnished to a more restricted composition than the A.I.S.I. range. For instance, the following specification limits were set forth by two leading manufacturers of seamless tubing:

	Maker A	Maker B
Carbon	0.15% max.	0.20% max.
Manganese	1.00 max.	1.50 max.
Sulphur	0.030 max.	0.025 max.
Phosphorus	0.030 max.	0.025 max.
Silicon	0.75 max.	0.75 max.
Chromium	25.00/28.00	26.00/30.00
Nickel	—	1.00 max.
Nitrogen	0.10 min.	0.12/0.25

Minimum physical properties of cold drawn, fully annealed tubing are as follows:

Ultimate tensile strength	70,000 psi.
Yield strength	40,000 psi.
Elongation in 2 in. (full tube)	18%
Elongation in 2 in. (strip)	15%
Rockwell hardness	B-95 max.
Brinell hardness	207 max.

Similar properties may be expected in plate, sheet, strip and other wrought forms of the alloy, such as rod and wire, when in the fully softened condition. (See Table II, page 978.)

As currently manufactured, 25 to 30% chromium-irons usually contain a purposeful addition of nitrogen in the form of chromium nitride. The nitrogen is incorporated in the alloy during the melting period by adding special, high nitrogen ferrochromium. On the basis of experimental evidence obtained by The Babcock & Wilcox Tube Co. and other investigators, it may be stated that the chief benefit derived from the chemically combined nitrogen is from its ability to refine the

*Editor's Note: A complete list of the reports made to Rubber Reserve Co. on the metals research program is printed on page 1024. Eventually copies of all of these reports may be purchased from the Office of the Publication Board, Department of Commerce, Washington, D. C. Some of them may now be had from Rubber Reserve.

grain structure and to inhibit grain growth at elevated temperatures, thus making for better workability, both hot and cold. Inasmuch as experiments, conducted by us[55]* indicated that nitrogen additions in excess of 0.30% had a tendency to increase hardness and strength but to reduce ductility markedly, the limits of 0.12 to 0.25% nitrogen were adopted for tubing manufacture. The data in Tables III, IV (page 979), and V (page 980), obtained by the Electro Metallurgical Co.,[102] are cited to illustrate the effect of nitrogen on the physical properties of wrought and cast 25% chromium steels.

Table I — Current Specifications

	A.I.S.I. Type 446*	A.I.S.I. Type 446* (P-518-T Tubing)	A.S.T.M. A-268-44T† Tubing	A.S.T.M. A-176-44† Grade 6 (Plate, Sheet and Strip)
Carbon	0.35% max.	0.20% max.	0.20% max.	0.35% max.
Manganese	1.00 max.	1.00 max.	1.50 max.	1.00 max.
Sulphur	0.040 max.	0.04 max.	0.030 max.	0.040 max.
Phosphorus	0.040 max.	0.04 max.	0.030 max.	0.040 max.
Silicon	1.00 max.	0.75 max.	0.75 max.	1.00 max.
Chromium	23.00/27.00	23.00/30.00	23.00/30.00	23.00/27.00
Nickel	—	—	0.50 max.	0.60 max.
Nitrogen	—	—	0.10/0.25	—

*Steel Products Manual, American Iron & Steel Institute.
†A.S.T.M. Standards, 1944 revision.

The alloy has been used for its heat resistance as thermo-couple protection tubes, as soot blower elements in steam boilers, for retorts, baffles or other parts of heat treating furnaces and, according to Furman,[43] it has been used in cast form for grates, rollers and disks and in other furnace parts. A high carbon variety containing 2.0 to 3.0% carbon or more has been used in cast form for its combined heat and abrasion resistance in rabble blades and sintering furnace parts.[54] Notwithstanding its relatively poor high temperature strength, the high chromium-iron alloy is usually preferred to the chromium-nickel, heat resistant alloys for applications where appreciable quantities of sulphur are contained in the hot gases coming in contact with the metal.

As a corrosion resistant material, this high chromium-iron alloy has been used in pumps and pipe lines for handling seepage water in metal mines, for piping and heat exchange equipment handling nitric acid and nitrating solutions (to which it is highly resistant) — also as digester heater tubes handling solutions of salt and nitric acid. It also has been used, albeit to a limited extent, in handling or heating sulphite liquors (calcium bisulphite plus sulphurous acid) encountered in paper making, as well as for handling acetic and phosphoric acid in other chemical processes. A modification of the alloy, containing approximately 4% nickel and 1.5% molybdenum, has found application in sulphite process paper work and for piping in the sulphuric acid alkylation process; in these uses the molybdenum confers greater resistance to reducing acid conditions. In general, the alloy offers excellent resistance to most forms of oxidizing chemical environment but may fail rapidly in reducing acids such as sulphuric or hydrochloric.

The 27% chromium-iron alloy has not enjoyed wider use heretofore because of its relatively poor strength properties at elevated temperature, its tendency toward embrittlement on slow cooling through or on heating in the vicinity of 885° F., its notch sensitivity at the lower temperatures, and for certain other peculiarities which make fabrication difficult. Furthermore, many of the lower chromium-nickel austenitic steels offer ample corrosion resistance or heat resistance (or both) for applications in the process industries, and they may also be more readily fabricated, especially where welding is involved.

Table II — Mechanical Properties of Annealed Stainless, Type 446*
American Society for Testing Materials[113]

Form	Tensile Strength	Yield†	Elong. in 2 In.	Red. of Area	Izod Impact	Rockwell Hardness	Ductility	
							Erickson Mm.	Olsen In.
Sheet	85,000	55,000	25.0	—	—	B-83	7/8	0.275/0.375
Strip	87,000	55,000	25.0	—	—	B-84	7/8	0.3 /0.4
Plate	85,000	55,000	25.0	—	2	—	—	—
Tubing	83,000	55,000	25.0	50.0	—	B-85	—	—
Bars	85,000	55,000¶	25.0	45.0	2	B-84	—	—
Wire	90,000	50,000	25.0	—	—	B-84	—	—
	135,000‡	115,000‡						

*Nitrogen usually 0.10% min.
†Yield point by dividers.
‡Wire, cold drawn 65%.
¶Proportional limit: 37,000 psi.

*Numbers as superscripts refer to papers listed in the bibliography, pages 1004 and following.

According to Curme,[21] the high chromium-iron alloys have proven superior to the chromium-nickel alloys for service involving the thermal decomposition of hydrocarbons, as in the production of ethylene and propylene. Chromium-irons were found to be less subject to carbon decomposition in the heater tubes and had less catalytic effect on the contained gases. Carburization tests in natural gas, made by The Babcock & Wilcox Tube Co., demonstrated likewise that 27% chromium-iron offered greater resistance to carbon absorption than the austenitic stainless steels.[131]

Tests of catalytic activity, carried out by several oil and chemical companies, indicate that a chromium content higher than about 25% is necessary for maximum resistance to the alternate drogenation. Nevertheless, tests in plant service involving long exposure of 25% chromium, 20% nickel alloy tubing and other parts, indicated satisfactory performance of this alloy under dehydrogenation conditions.

The physical constants of 25 to 30% chromium-iron alloy and mechanical properties at room temperature are given in Table VI.

Properties at Elevated Temperatures

Preheaters, lines, valves, catalyst tubes or vessel reactors in the dehydrogenation process operate at elevated temperatures, hence the high temperature strength properties of 27% chromium-iron alloys are of importance in the design and performance of plants where this metal is used. Its high temperature properties are also of interest when using the alloy for thermal cracking, as in the production of ethylene, propylene and butylene, for handling organic acid vapors, and for other high temperature applications.

In dehydrogenation, the gas temperature at the reactors ranges from about 1050 to 1250° F., while during the regeneration cycle, the temperature at the start may be dropped to 800 to 950° F. to compensate for the heat produced when the carbon is

Table III — Physical Tests After Treatment at Elevated Temperatures
Electro Metallurgical Co.[102]

Composition‡			Condition of Metal	Yield Point	Tensile Strength	Elong. in 2 In.
Cr	C	N_2				
25.50★	0.14	0.05	Heated at 1605° F. 8 hr. and water quenched	54,350	75,600	35%
25.50†	0.14	0.05	Heated at 1605° F. 8 hr. and water quenched; held 7 days at 2010° F. and air cooled	None shown	39,100	0
25.08†	0.15	0.22	Heated at 1605° F. 4 hr. and air cooled	50,250	78,150	31
25.08†	0.15	0.22	Heated at 1605° F. 4 hr. and air cooled; held 7 days at 2010° F. and air cooled	58,250	83,600	34

★¼-in. plate. †⅞-in. plate.
‡These steels contain normal percentages of silicon and manganese.

reducing and oxidizing conditions encountered in catalytic dehydrogenation. Therefore, tubing and other parts which are in contact with heated gases or dehydrogenation reaction products, are preferred with a minimum chromium content of 26%.

Tests made in our laboratory on a variety of weld metals and chromium alloy compositions revealed that nickel in excess of 10% tends to depreciate the chemical inertness of nickel-bearing alloys in butane dehy-

Table IV — Physical Tests on High Nitrogen and Low Nitrogen Steels Containing 25% Chromium[102]

Composition‡			Condition of Metal	Yield Point	Tensile Strength	Elong. in 2 In.	Brinell Hardness
Cr	C	N_2					
26.19★	0.15	0.07	Hot rolled	52,600	79,300	16%	172
25.08†	0.15	0.22	Hot rolled	83,000	98,300	18	217
26.19★	0.15	0.07	Heated at 1605° F. 8 hr. and water quenched	53,250	76,500	34	159
25.08†	0.15	0.22	Heated at 1650° F. 4 hr. and water quenched	50,800	78,150	31	166
26.19★	0.15	0.07	Heated at 1740° F. ½ hr. and air cooled	46,800	69,900	6	159
25.08†	0.15	0.22		53,000	78,400	33	179
26.19★	0.15	0.07	Heated at 1920° F. ½ hr. and air cooled	58,300	67,700	3	166
25.08†	0.15	0.22		52,400	77,300	34	174
26.19★	0.15	0.07	Heated at 2100° F. 15 min. and water quenched	72,000	80,800	2	207
25.08†	0.15	0.22		81,100	105,600	38	217
25.30★	0.19	0.24	Heated at 1605° F. 4 hr. and furnace cooled	62,900	91,600	24	212

★¼-in. plate. †⅞-in. plate.
‡These steels contain normal percentages of silicon and manganese.

burned from the catalyst. Temperatures during burn-out may rise to 1350° F. and at times probably higher. Pressures are low, not exceeding about 50 psi. inlet on the gas and 150 psi. inlet during the regeneration cycle.

In the other processes, metal temperatures may extend to 1500° F., or even higher with low pressures.

The 25 to 30% chromium alloys have been used chiefly for their resistance to scaling; in service involving scaling conditions, the parts remain under stress for a long period of time. Creep and rupture strength are therefore of greater interest than the short-time tensile properties. The latter are of interest, however, in indicating the effect of temperature on properties and for indicating ductility in certain fabricating operations.

Short-Time Tensile Properties — Short-time tensile properties for annealed material containing about 30% chromium are given in Table VII. The test pieces were held one hour at temperature and pulled at a rate of 0.05 in. per min. Supplementing these data, high temperature static tensile properties were determined by Babcock & Wilcox[135] during the course of investigations for Rubber Reserve Co. on three heats intended for tubing manufacture. The data obtained on specimens from annealed 1-in. rounds are given in Fig. 2. Compositions of the three heats tested are shown in the caption. The strain diagrams from

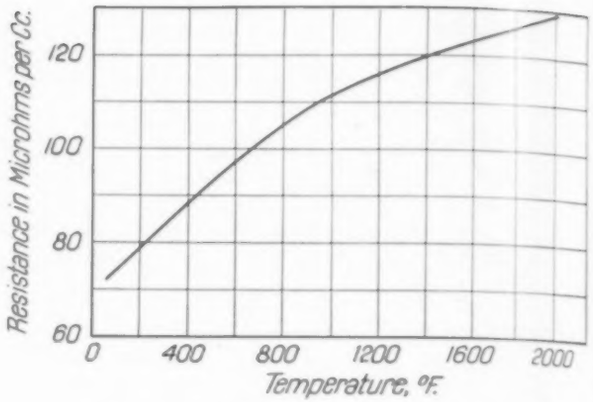


Fig. 1 — Electric Resistance of 28% Chromium Irons at Various Temperatures[67]

Table V — Tests on High Nitrogen, Chromium Steel Castings

Size treated: 1 in. diameter. Size tested: 0.505-in. diameter by 2.2 in. Electro Metallurgical Co.[102]

Composition				Condition of Metal	Yield Point	Tensile Strength	Elong. in 2 In.	Transverse Test		Brinell Hardness
Cr	C	Ni	N_2					Breaking Load	Deflection	
24.21	0.32	0.15	0.20	As cast	63,000	82,000	3	7,800 lb.	0.30 in.	202
24.21	0.32	0.15	0.20	Heated at 1560° F. 2 hr., furnace cooled to 1110° F., then air cooled	52,200	81,400	15	—	—	179
24.43	0.35	1.20	0.26	As cast	76,600	96,500	3	8,700	0.53	223
24.43	0.35	1.20	0.26	Heated at 1560° F. 2 hr., furnace cooled to 1110° F., then water quenched	61,000	103,000	6	—	—	200
24.85	0.42	1.51	0.22	As cast	—	—	—	9,700	0.60	223
25.97	0.32	1.13	0.30	Heated at 1560° F. 2 hr., furnace cooled to 1110° F., then water quenched	62,700	106,400	7	—	—	207
27.35	0.56	0.34	0.32	As cast	50,000	75,000	2	6,800	0.23	196
								Red. of Area	Average Izod Impact	
24.74	0.14	3.11	0.06	As cast	71,000	95,000	8	10.0%	6 ft-lb.	196
24.97	0.13	3.07	0.185	As cast	65,000	101,000	17	17.0	5	193
24.74	0.14	3.11	0.06	Held ½ hr. at 1920° F. and air cooled	65,600	90,400	23	36.7	11	179
24.97	0.13	3.07	0.185	Held ½ hr. at 1920° F. and air cooled	67,000	97,000	38	47.2	45	187
26.0	0.25	—	Low	Heated 6 hr. at 1650° F. and air cooled	48,000	58,000	1.0	1.1	1.0	153
25.50	0.30	—	0.20	Heated 6 hr. at 1650° F. and air cooled	52,000	82,500	15	20	5	163

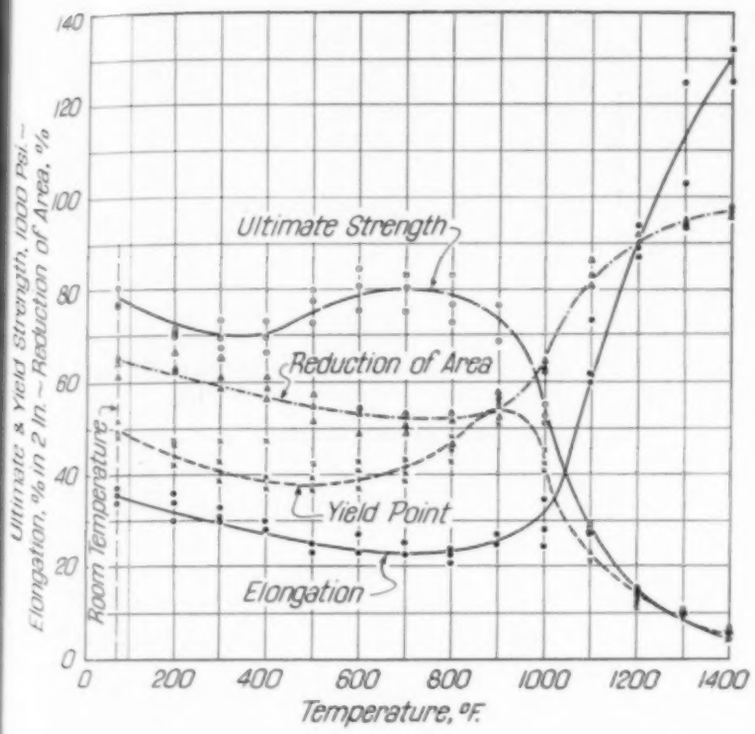


Fig. 2 — Short-Time Tensile Properties of Three Heats of Annealed 27% Chromium-Iron Intended for Tubing Manufacture. (The Babcock & Wilcox Tube Co.[135])

Heat No.:	21881	24162	11358
Carbon	0.15	0.196	0.22
Manganese	0.80	0.73	1.12
Silicon	0.48	0.54	0.71
Chromium	27.21	27.31	28.12
Nickel	0.18	0.16	0.26
Nitrogen	0.14	0.15	0.13

which the yield points were taken in these tests were of interest as they showed the characteristic "slip-step" straining system at certain temperatures, indicative of precipitation hardening or aging phenomena.

Average mechanical properties at high temperature, compiled from several sources by A.S.T.M.,[113] are given in Fig. 3 (page 982). These data may be considered as typical for the alloy although some variation may be expected depending on specific composition and original condition of the metal. Tensile strength is maintained fairly well up to about 1000° F. but ductility decreases; above this temperature, the strength drops rapidly and ductility improves considerably. The short-time tensile properties of cast 30% chromium steel are somewhat similar to the wrought metal, as may be noted from Fig. 4.

Creep strength data for wrought 25 to 30% chromium-iron are relatively meager and are not consistent, although they indicate that the alloy has poor strength properties especially at temperatures above 1000° F. White and Clark[54] report stresses for various rates of creep for 27% chromium, 0.20% carbon alloy at 1200° F. as follows:

Creep Rate in 1000 Hr. at 1200° F.	Stress
1.0%	2700 psi.
0.5	2100
0.1	1300
0.05	1200
0.01	1000

The data listed at the top of page 982 are reported by Muller-Berghaus[91] for 28% chromium steel. The stress is for a creep rate of 0.0001% per hr., after a permanent elongation of 0.2%.

Table VI — Physical Constants and Room Temperature Mechanical Characteristics

Specific gravity[113]	7.6 g./cm.³ (0.27 lb./in.³)
Specific volume	0.1305 cm.³/g. at 20° C.
Specific heat	0.12 B.t.u./lb./°F. (32 to 212° F.)
Electrical resistance	67.0 microhms/cm.³ (see also Fig. 1)
Thermal conductivity[47, 91]	0.050 cgs. units at 100° C. 0.058 cgs. units at 500° C.
Thermal expansion	0.0000059 in./in./°F. (32 to 212 F.) 0.0000063 in./in./°F. (32 to 1000 F.) 0.0000076 in./in./°F. (32 to 1832 F.)
Modulus of elasticity	29,000,000 psi.
Melting range[113]	2700 to 2750° F. (1482 to 1510° C.)

Average Mechanical Properties	Forged or Rolled Sections (Annealed)[113]	Cold Worked[90]
Ultimate stress, psi.	70,000/100,000	85,000/175,000★
Yield point, psi.	40,000/ 65,000	55,000/155,000★
Elongation, % in 2 in.	25/ 35	25/ 2★
Reduction of area, %	45/ 65	55/ 25★
Brinell hardness	160/200	150/250★
Endurance limit in air (10^7 cycles)	44,000 psi.†	

★Range depends on amount of cold work applied.

†Endurance limit cited[19, 20] is for a steel containing 0.20% C, 27.37% Cr (low Si and Mn) and possessing the following properties in the annealed state: 81,000 psi. ultimate, 44,500 psi. yield, 28% elongation in 2 in., 57% reduction of area, Charpy impact 2.0 ft-lb.

Creep Strength of 28% Cr Steel[91]

Temperature	Stress for Creep Rate of 0.0001% per Hr.
1100° F.	5100 psi.
1200	3600
1290	2600
1380	1600
1470	1000
1650	300

Norton[26] tested a steel containing 0.20% C, 0.80% Mn, 0.36% Si, 0.51% Ni, and 26.94% Cr, and obtained the following creep values:

	1200° F.	1350° F.
1% in 10,000 hr.	1600 psi.	480 psi.
1% in 100,000 hr.	1000	180

Norton's data together with information obtained from Allegheny Ludlum Steel Corp.[67] are plotted in the composite creep curves shown in Fig. 5, which are believed to be reliable and satisfactory and are recommended for use in plant design. Stresses for rates of 0.01% per 1000 hr. (1% per 100,000 hr.) and 0.1% per 1000 hr. (1% in 10,000 hr.) are plotted. Table VII gives data found for 30% Cr alloy by Allegheny Ludlum. Other data from the A.S.T.M. compilation[113] and from other sources as reported by Hoyt[117] are recorded in Table VIII.

Creep strength for chromium steel castings containing 0.5 to 2.0% C, 1.0 to 1.5% Si, 27.0 to 30.0% Cr, as given by Schulte[114] and measured on the basis of 1% permanent elongation in 1000 hr., are as shown in the little box on page 983.

Table VIII — Creep Strength of Stainless Steel, Type 446

Temperature	1% in 10,000 Hr.		1% in 100,000 Hr.[117]
	A.S.T.M.[113]	Hoyt[117]	
900° F.	17,000	17,000	13,000
1000	6,500	6,000	4,500
1100	3,000	2,700	2,000
1200	1,500	1,200	800
1300	600	400	300
1400	300	200	150

Table VII — Short-Time Tensile Properties at Elevated Temperatures★

Temperature	Tensile Strength	Elongation in 2 In.	Reduction of Area
Room	93,480	26.0%	60.0%
600° F.	83,170	19.0	53.0
800	82,380	19.0	48.0
900	80,630	20.0	42.0
1000	70,520	15.0	47.0
1100	39,280	47.0	73.0
1200	24,210	47.0	85.0
1300	10,860	57.0	91.0
1400	6,990	80.0	93.0
1500	4,245	133.0	98.0
1600	3,355	165.0	99.0

★Chemical analysis of steel investigated: 0.10% C, 0.74% Mn, 0.021% P, 0.014% S, 0.131% Si, 30.01% Cr, 0.570% Ni. (Allegheny Ludlum Steel Corp.[67])

Creep strength for 28% chromium-iron castings, collected by one investigator are reported by Hoyt[117] as follows:

Temperature	Creep in 1000 Hr.		
	0.01%	0.10%	1.00%
1400° F.	1000	1250	1500
1500	600	750	1050
1600	375	450	600
1700	250	350	500
1800	140	200	300

Rupture Strength — Rupture tests on wrought 27% chromium-iron have been made by R. H. Thielemann of General Electric Co. at 1100° F.

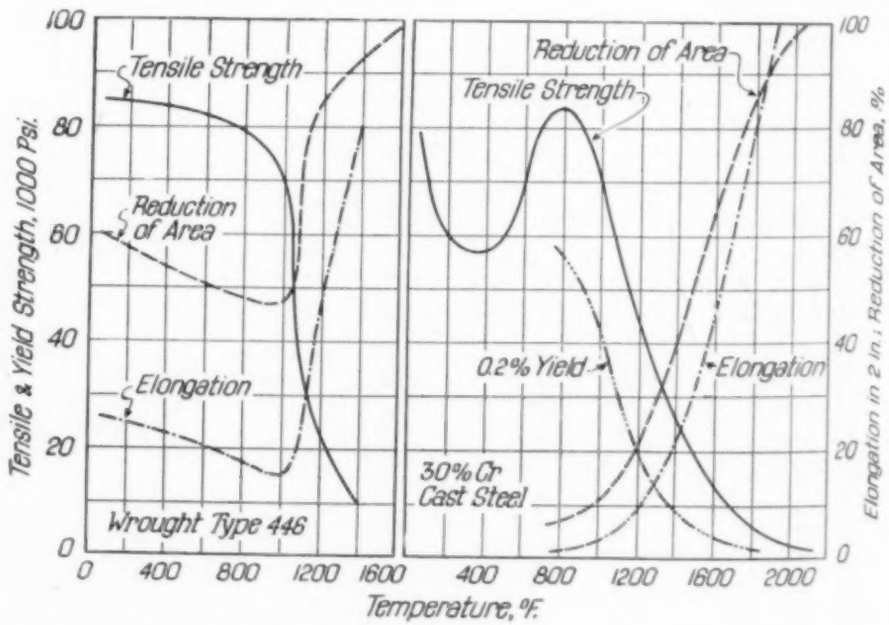


Fig. 3 and 4 — Short-Time High Temperature Tensile Properties of 23 to 30% Chromium Wrought Steel, Type 446, and 30% Chromium Cast Steel, Respectively. (American Society for Testing Materials[113], and Schulte[114])

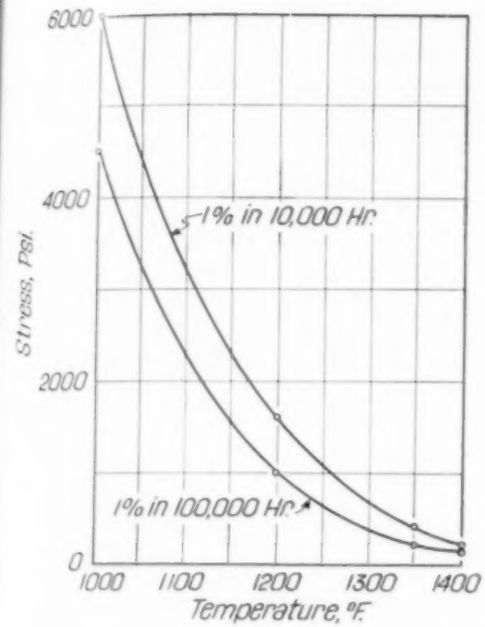


Fig. 5 — Recommended Creep Values for Design. Composite data from Norton[23] and Allegheny Ludlum Steel Corp.[67]

on an alloy containing 0.16% C, 0.74% Mn, 0.376% Si, 0.36% Ni and 26.62% Cr. Mr. Thielemann informed me that the value found for rupture in 1000 hr. at 1100° F. was 5900 psi.; for rupture in 10,000 hr. it required 3750 psi.; and for rupture in 100,000 hr., 2400 psi.

Timken Roller Bearing Co.[109] also reported rupture strength on a steel of the following composition: 0.12% carbon, 0.45% manganese, 0.47% silicon, 0.009% sulphur, 0.014% phosphorus, 25.40% chromium and 0.125% nitrogen.

Their tests at temperatures ranging from 1200 to 1800° F. are plotted to logarithmic coordinates in Fig. 6. At 1200 and 1400° F. a straight line relationship exists between stress and time for rupture, while at the higher temperatures a break occurs at about 10 hr. The results show that the 27% chromium-iron alloy possesses very low strength at these temperatures, which is confirmed by the low short-time tensile and creep strength, as noted earlier in this section. The ruptured specimens of 27% chromium-iron, even on the most prolonged tests, exhibited a high degree of ductility, the elongation values ranging from 55 to 110%. Figure 7, also from Timken data, shows the rupture values of this steel for definite time to fracture, plotted against temperature.

In summary, it may be stated that the 25 to 30% chromium-iron alloys show relatively poor strength properties at temperatures exceeding 1000° F. Ample sections should be employed in design to prevent over-stressing, and adequate support in furnace installations to prevent sagging.

Impact Properties and Notch Sensitivity

Krivobok[56] has shown, Fig. 8, that the resistance to impact at room temperature of a series of iron-chromium

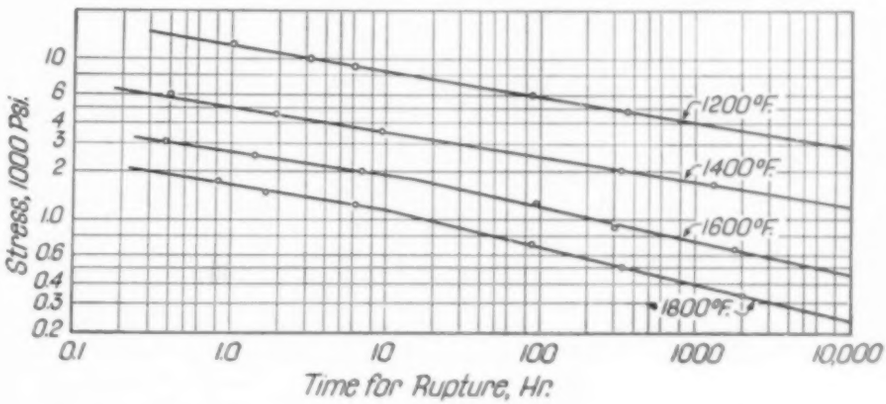


Fig. 6 — Logarithmic Plot of Stress-Rupture Properties of 27% Chromium-Iron Containing 0.12% Nitrogen. (Timken Roller Bearing Co.[109])

27 to 30% Cr[114]	
Temperature	Creep Strength
1110° F.	5000 psi.
1290	1100
1470	430
1650	170
1832	70
2010	40

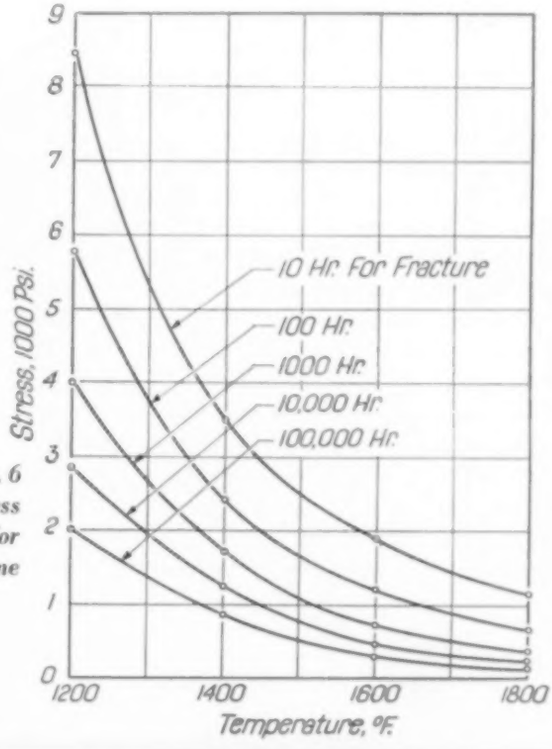


Fig. 7 — Data of Fig. 6 Replotted to Show Stress and Temperature for Rupture in Given Time

alloys sharply declined when the chromium content exceeded about 15%. This behavior was found to be independent of carbon content and heat treatment in the lower carbon alloys, and it was not reflected in the static tensile properties. (Similar results were obtained by Hodge[54] for welded specimens of chromium steel.) The 25 to 30% chromium-iron alloys are similar to the lower chromium-iron alloys in this respect and almost invariably show low impact resistance under standard notched-bar forms of testing — that is, Izod and Charpy. Typical values for impact generally show from 1 to 4 ft-lb. at ordinary temperature, irrespective of grain size, size of section, heat treatment or minor variation in composition. This statement is confirmed by values reported by MacQuigg[54] and others.[103]

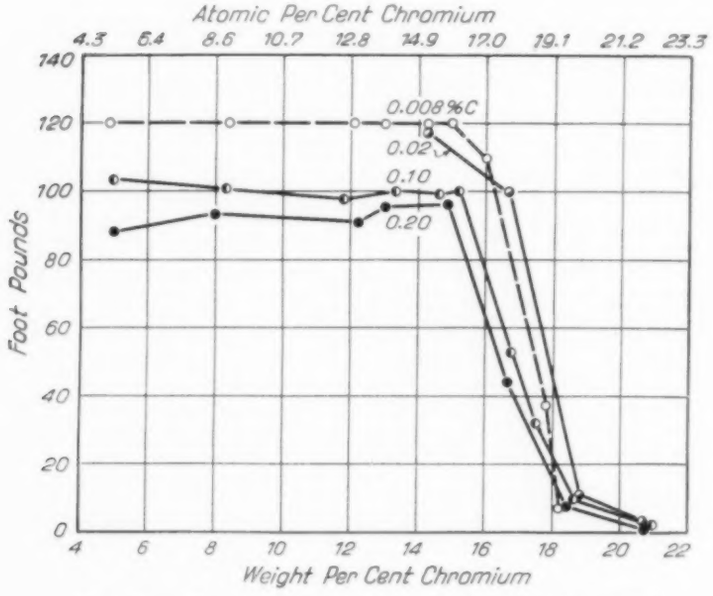


Fig. 8 — Energy Absorbed by V-Notched Standard Charpy Specimens of Chromium-Iron Alloys Ranging in Carbon From 0.008 to 0.20% (Krivobok[56])

Although there are not sufficient data to construct a complete temperature-composition curve, it can be stated nevertheless that the transition temperature from brittle to tough condition is below room temperature for 13% chromium steel and above room temperature for 18% chromium steel. It may be inferred therefrom that 27% chromium steel becomes tough when heated to a sufficiently elevated temperature. Consequently, when adjudged merely from impact tests at room temperature, the alloy would be considered too brittle for service involving impact stresses but would be suitable for elevated temperature service. This has been verified by practical experience providing suitable precautions are taken, as is pointed out definitely in the summary and conclusions on pages 1002 to 1004.

Eberle[126] and Fowler[124] have shown that toughness in 27% chromium-iron alloy is much improved by heating to 300° F.; in the weld metal of high nitrogen content the impact resistance rose from 1.5 ft-lb. Charpy average at room temperature to 16.5 average at 300° F. Likewise, Riedrich and Loib[103] showed that toughness is improved considerably in annealed metal on heating to 300° F. Even after embrittlement from the formation of sigma phase, or after embrittlement by heating at 885° F., there will probably be some improvement in toughness with increased temperature. It is rather likely that a considerable loss in room temperature or basic impact resistance will result from the factors just mentioned so that higher temperatures will be required to restore reasonable toughness in such embrittled metal.

While the specific effect of nitrogen is to refine the grain structure and improve static ductility, it does not appear to be of appreciable help in improving impact properties of 27% chromium-iron alloy. However, when used in conjunction with 1 to 3% nickel, nitrogen appears to have considerable value in this respect, as has been shown by Franks[55] and Colbeck and Garner.[77] This is especially true when such metal is quenched from a high temperature (1920 to 2100° F.), whereupon the nickel and nitrogen tend to retain a considerable amount of tough austenitic phase in the microstructure. (In low nickel alloys the structure consists essentially of chromium ferrite and carbides.) Small amounts of nickel have been added to electrode materials to produce additional toughness in high chromium weld metal; the austenitic phase is created in the weld metal and is retained on cooling from welding heat so that toughness is improved.

Notch Sensitivity

The 25 to 30% chromium alloys are extremely notch sensitive, as has been indicated by values obtained in notched-bar impact tests. This was further demonstrated recently in the laboratory of The Babcock & Wilcox Tube Co.,[139] where some properly annealed tubes of ½-in. wall thickness were machined to standard size 0.394 x 0.394-in. Charpy impact specimens. Without a notch, such specimens remained unbroken after absorbing 230 ft-lb. of energy. Specimens notched with a shallow saw cut, 0.005 in. deep, showed 72.0 ft-lb., whereas specimens with the standard form of

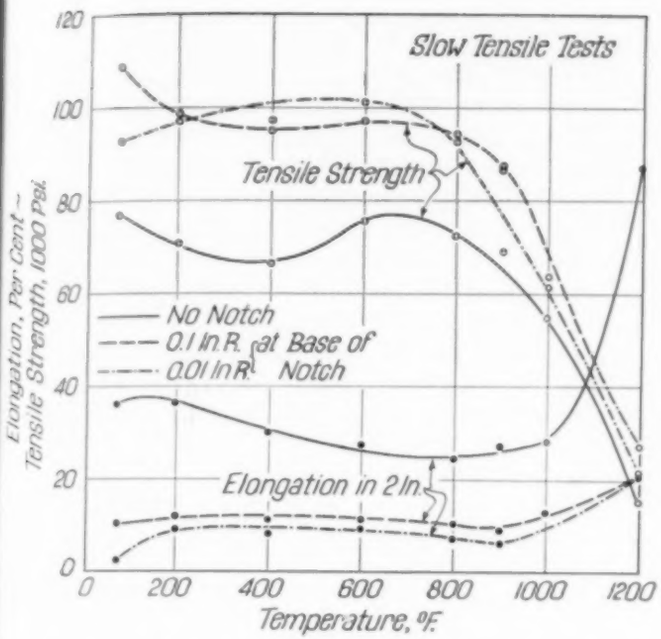


Fig. 9 — Effect of a Notch on the Short-Time, High Temperature, Tensile Properties of 27% Chromium-Iron. (The Babcock & Wilcox Tube Co.[139])

drilled keyhole notch broke at the usual 2.0 ft-lb. Eberle[127] performed similar experiments with welded specimens and found similar results for notched and unnotched specimens. Bagsar (in "Metallurgical Investigation for Butane Dehydrogenation Applications", dated Sept. 20, 1943) also confirmed these findings when he obtained 195 ft-lb. on unnotched 10-mm. square Charpy specimens of 27% chromium-iron alloy, and the usual 2 ft-lb. on the same material with standard keyhole notch.

Other practical demonstrations of notch sensitivity have been observed in 27% chromium-iron alloy in failure on deformation of tubes beveled for welding, failure in scratches at surface defects, guide marks and roll marks, and at threads and re-entrant angles in rolled or machined articles. Smooth surfaces, such as are obtained by cold drawing or cold rolling, are desirable to minimize failure due to shock stress or during deformation. Notch sensitivity is decreased by raising the temperature to 300° F. or higher and this should be done as a precaution where any liability to failure exists at ordinary temperature. Designs should be simple, in structures made of this alloy, and should avoid key-ways, re-entrant angles and sharp radii. The material should be as free of surface marks, scratches or other forms of notches as is consistent with present commercial manufacturing practice.

Recent and more detailed studies of notch sensitivity have been made by The Babcock & Wilcox Tube Co.[141] These included ordinary tensile tests at room and elevated temperatures on notched tensile specimens, as well as on notched-bar specimens tested in impact. Comparisons were made of the influence of notches of 0.10-in. and 0.01-in. radius versus unnotched specimens; the notches were of 45° V form, 0.06 in. deep. Diameter of specimens was 0.505 in. at the root of notch for those specimens which were notched, and a similar sized unnotched specimen was used in the comparison tests.

The tensile tests are shown in Fig. 9. The specific effect of the notch was to increase the tensile strength and decrease the elongation. This occurred throughout the temperature range employed. Little difference was found in the effects of the two types of notches, although a decrease in radius at the base of the notch resulted in consistently lower elongation values in the tensile test.

On the notch bend or impact tests, the

Table IX — Effect of Depth of Notch on Impact Strength*
The Babcock & Wilcox Tube Co.[139]

Temperature	Full Notch	⅔ Notch	⅓ Notch	No Notch
Heat 24162, 0.196% C, 0.73% Mn, 0.54% Si, 27.31% Cr, 0.15% N₂				
70° F.	8 ft-lb.	9 ft-lb.	15 ft-lb.	175 ft-lb.
200	50	69	119	170
400	78	91	120	145
700	72	82	106	118
800	70	77	115	112
900	61	72	98	121
1000	62	65	96	113
Heat 21881, 0.15% C, 0.80% Mn, 0.48% Si, 27.21% Cr, 0.14% N₂				
70	3	7	16	185
200	21	22	27	184
400	72	75	77	174
600	74	84	90	152
800	67	68	70	142
885	59	63	70	139
1000	48	60	73	129
1200	53	—	—	127
1400	61	—	—	110
1600	78	—	—	87

*Full notch of V form conforming with A.S.T.M. Specification E23-41T; ⅔ and ⅓-depth notches were produced by machining the notched side of the specimens to approximately ⅔ and ⅓ of the usual dimension. The specimens with no notch had the notch fully removed by machining to 0.315 in. thickness, thus leaving the area of the specimens at the base of the notch constant in all tests.

standard 45° V notch was employed with 0.010-in. radius at its base. The depth of the notch was varied from standard depth to none. All specimens were annealed and of identical composition. Notch sensitivity appeared to vary in individual heats, as may be noted by reference to Table IX. Recovery of toughness in notched specimens begins at moderately elevated temperature, but even so there still exists a decided difference in toughness between notched and unnotched bars. The wide difference in energy absorbed between unnotched specimens and those having a full V notch may be noted from Fig. 10. The value of preheating to reduce notch sensitivity is therefore apparent.

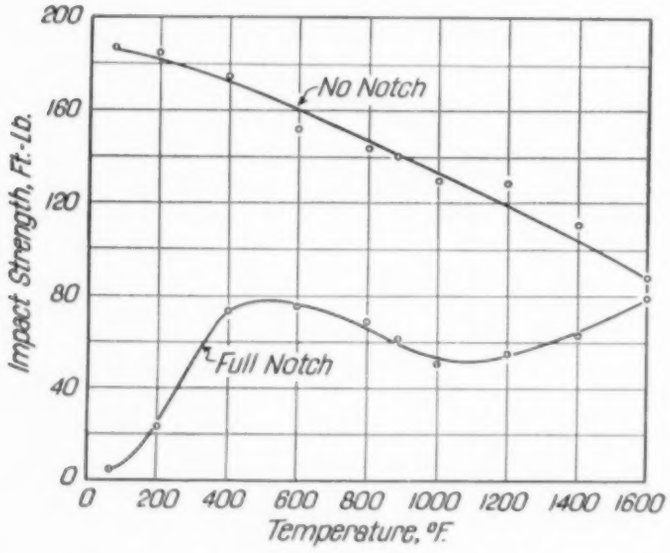


Fig. 10 — Effect of Temperature on the Impact Properties of 27% Chromium-Iron. No notch vs. full notch; specimen described in footnote to Table IX. (The Babcock & Wilcox Tube Co.[139])

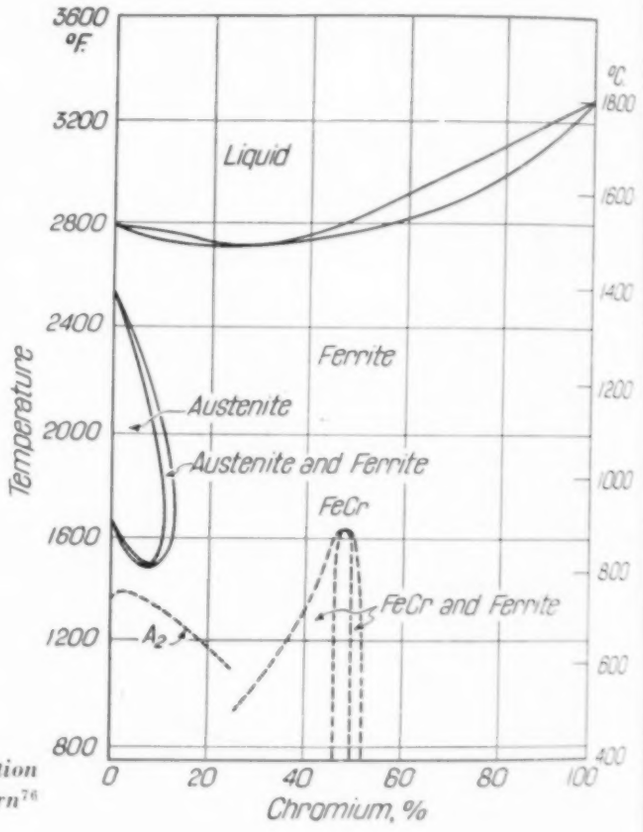


Fig. 11 — Iron-Chromium Constitution Diagram According to Bain and Aborn[76]

Structural Characteristics

The constitution of iron-chromium alloys has been studied by several investigators, including Adcock,[33] Bain and Griffiths,[18] Westgren,[23] Wever and Jellinghaus[38] and others.[12, 65] A typical constitution diagram of these alloys, as offered by Aborn and Bain,[76] is reproduced in Fig. 11. Lately, the region around 50% chromium has been intently studied.

In the range between 25 and 30% chromium, with the usual carbon content present in wrought alloys (about 0.25% max.) the commercial alloys are without appreciable allotropic transformation from about 1400° F. up to their melting point. A secondary constituent identified as the intermetallic compound sigma phase (marked FeCr in Fig. 11) may precipitate on long heating at intermediate temperatures.[65]

The ferritic alloys containing more than 15% chromium cannot become austenitic except in the presence of a considerable amount of austenite-forming elements such as carbon, nitrogen, nickel or manganese; low carbon chromium-iron alloys fall outside the area of the gamma loop (marked "austenite" in Fig. 11) and therefore can be hardened only by cold working. Heating ferritic alloys to elevated temperatures promotes grain growth and coarsens the structure, this effect being especially pronounced at temperatures above 1650° F. Recrystallization and grain refinement in unstrained metal do not occur on heat treatment in the usual sense but only after cold working.

A brittle, coarse grain is associated with the alloy in its cast state and in weld deposits. This is in contrast to a much finer grain resulting from forging or hot rolling. Consequently, mechanical working assumes unusual importance to the 27% chromium-iron alloy, as there is virtually no other means of controlling the structural characteristics and the related mechanical properties. As already

mentioned, nitrogen additions effectively inhibit grain growth at elevated temperatures. Decarburization of the surface layers under certain conditions of exposure at high temperatures will produce a coarse columnar structure, which appears as a sharply defined zone of coarse grains growing inwardly from the surface, and which detract from the ductility of the metal.

Cold working, following hot working operations, provides additional ductility and tends to smooth the surface, thus reducing or eliminating surface marks or notches to which the alloy is extremely sensitive — especially at temperatures below about 250° F. Cold working, which may be applied on rod, tube, sheet or wire, tends to break up and refine the microstructure, especially when followed by an appropriate heat treatment, and this combination of working and annealing appreciably improves ductility. Initial passes should be light to avoid breakage until the alloy has become "conditioned"; heavier working may then be attempted. Moderate warming of the article before deformation is usually quite helpful.

In the annealed or softened state, the structure of the alloy consists of carbides and chromium ferrite, the grain size being dependent on the amount of work applied to the section. On heating above 1700° F. a small amount of austenite may be formed in the vicinity of carbide or nitride particles, the amount depending on the percentages of these elements present in the alloy, but usually too small to cause appreciable hardening even on quenching.

It appears that the intermetallic compound sigma phase, usually assigned the formula FeCr, is formed in alloys containing more than 25 and less than 55% chromium. The rate of transformation of ferrite to this brittle phase is extremely slow in pure iron-chromium alloys unless they have been previously cold worked, but its formation is accelerated in the presence of a few per cent of elements such as nickel, manganese and silicon. The rate of formation is also the more rapid the nearer the temperature is maintained to its upper limit of stability (1600° F.). The sigma constituent, when developed in mass, appears to be converted to ferrite at temperatures above about 1625° F. Conversion occurs at lower temperatures when sigma constitutes only part of the alloy — that is, when it is present as a mixed phase with ferrite.

Becket[68] concluded that the brittleness which develops in commercial iron-chromium alloys at 885° F. is not related to sigma phase formation but is more likely due to the presence of some other submicroscopic precipitate of unknown composition. Further study of the nature of this brittleness occurring at 885° F. is necessary. Becket suggested the use of polarized light after etching in 25% hydrochloric acid in alcohol for differentiation between carbides, ferrite, and the sigma phase under the microscope.

Sigma phase is extremely hard and brittle and is non-magnetic. Several investigators offer evidence for the existence of this intermetallic compound. Bain and Griffiths[18] reported sigma phase constituent in 27% chromium-iron tubing after several months' exposure at 1020° F., and have verified its presence by X-ray diffraction analysis. It was reported that heating for several hours at 1470° F. or even 30 min. at 1560, entirely removed the brittleness due to this precipitated phase. Sigma transformed to ferrite.

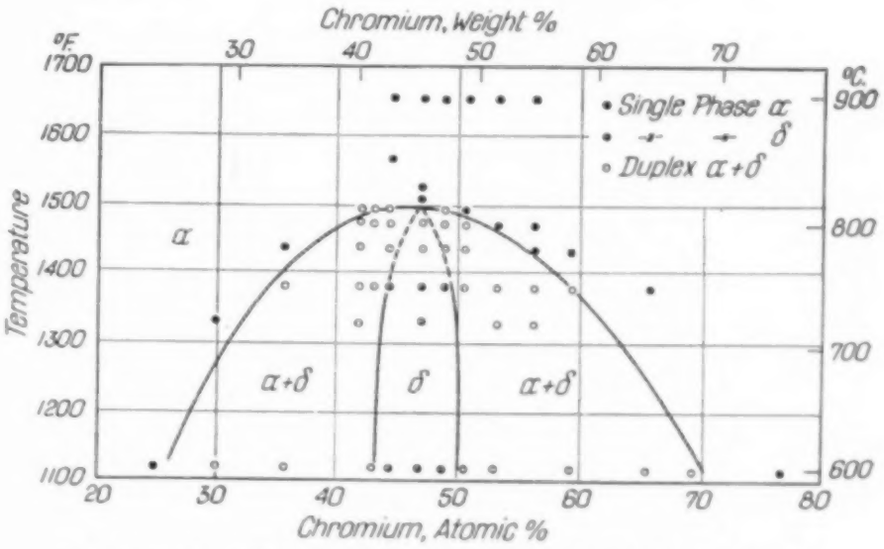


Fig. 12 — Iron-Chromium Phase Diagram in Binary Alloys of High Purity; 25 to 76% Chromium (Cook and Jones[115]). This diagram shows pure sigma phase from 44 to 50% chromium and mixed alpha and sigma phase from about 25 to 44% of chromium with phase boundaries for temperature interval of 1100 to 1500° F.

Although Adcock[33] was unable to ascertain its presence in his purest iron-chromium alloys, sigma was positively identified by Cook and Jones[115] who worked on Adcock's high purity iron-chromium samples (down to about 25%

chromium) after long annealing at 1110° F. Their modification of the phase diagram, Fig. 12, indicates that a mixed structure of alpha phase (chromium ferrite) and sigma phase (FeCr) can occur in the temperature range between about 1110 and 1240° F. in iron alloys containing 25 to 30% chromium. (It should be pointed out that variation of composition with respect to manganese, silicon or nickel in the specific alloy may extend or otherwise modify this range.)

The Babcock & Wilcox Tube Co. in the course of various investigations on high alloy steels after certain thermal treatments or services, have noted sigma phase in varying amounts in such alloys as 27 Cr, 4 Ni, 1½ Mo; 25 Cr, 12 Ni; 25 Cr, 15 Ni, 2 W; 25 Cr, 20 Ni; and also in the 27% chromium-iron alloy.

The representative microstructure of 27% chromium-iron, after exposure for 131 days at 1000 to 1050° F. is shown by Fig. 13. This structure is composed of chromium ferrite and spheroidized carbides, while a third constituent, never found in annealed metal, forms a continuous network around the grains. This constituent is shown more clearly at higher magnification in Fig. 14, where a light, 2-sec. etch shows it to be a structureless phase; few of the familiar chromium carbides have as yet made their appearance, indicating that the third, or sigma phase, constituent is more soluble in the etching reagent than the carbide phase. Continued etching, as shown by Fig. 15, results in blackening or eating out of the sigma phase network and outlining the carbides within the center of the grains. While the presence of sigma phase is readily disclosed when it occurs in such massive form, the fact that it is preferentially etched is an aid in detecting its presence when it occurs in smaller quantities.

Bain and Griffiths,[18] in their investigation of chromium-iron and chromium-nickel-iron alloys, showed that the sigma constituent was non-existent above 1470 to 1740° F., the exact temperature varying with composition, and that heating above such a critical temperature develops chromium-ferrite that persists in a specimen cooled at an average rate, but that a few hours of heating below the critical temperature range for the specific alloy develops the sigma phase. Decreasing the alloy content — chromium or nickel or the total of both — lowers the critical temperature and increases the time required for sigma phase to form. Heating for several months at 1025° F. was required to develop sigma phase at the grain boundaries of a 30% chromium-iron alloy. Jette and Foote,[60] using the X-ray method to investigate this compound in a 50% chromium-iron alloy, developed sigma phase after long heating at temperatures ranging from 1110 to 1425° F. Both groups of

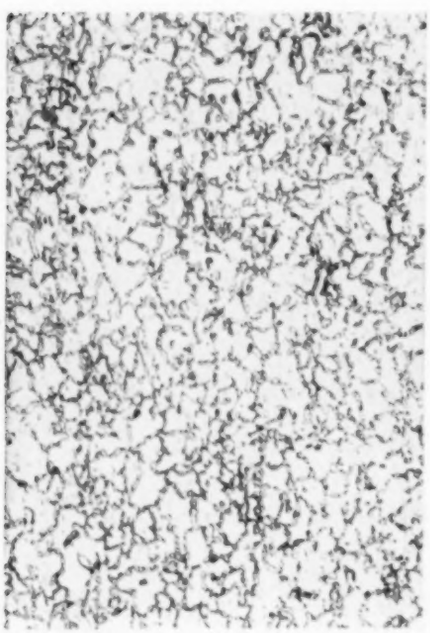


Fig. 13 — 27% Chromium-Iron Tube Showing Ferrite, Carbides and Intergranular Sigma Phase Constituent Formed After Heating 131 Days at Approximately 1050° F. Etched in aqua regia. 200×

Fig. 14 — Same as Fig. 13, Repolished and Etched 2 Sec. in Aqua Regia. This photograph shows sigma phase with some small spheroids of carbide around which the sigma phase has formed. 1000×

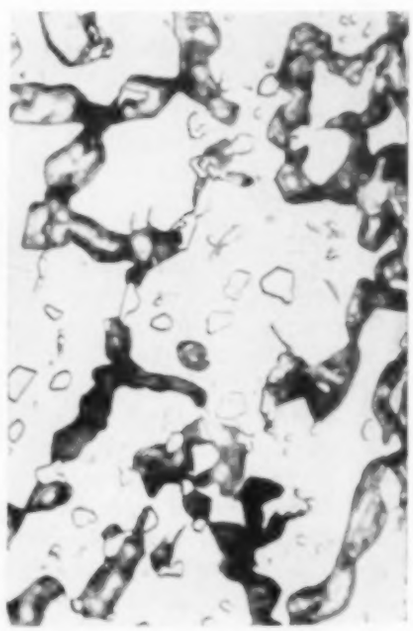


Fig. 15 — Same Specimen Shown in Fig. 13 and 14, Etched 10 Sec. in Aqua Regia During Which the Sigma Phase Is Blackened and Eaten Out. Carbides appear mainly in center of ferrite grains. 1000×

investigators show that the formation of sigma constituent is accompanied by a pronounced increase in hardness of the alloy.

In the light of these investigations, it may be inferred that sigma phase will form in 25 to 30% chromium-iron alloys on long heating within a range of temperature from about 1025 to 1300° F. The amount formed will depend on the composition of the specific alloy with respect to chromium, silicon, manganese and nickel, and the rate of formation will be accelerated if the metal has been cold worked. According to Bain,[18] carbides do not influence the formation of sigma phase but augment the volume of austenite in the alloy. Bradley and Goldschmidt[98] stated that nitrogen plays no part in the sigma phase transformation, which may occur in the absence of nitrogen. However, Becket[68] pointed out that nitrogen tends to accelerate transformation to sigma phase in alloys containing more than about 35% chromium.

The formation of sigma phase is accompanied by an increase in hardness, and it is known that other physical properties of the metal are affected to the detriment of its ductility and toughness. Changes caused by sigma phase will likely be more noticeable when the alloy is at temperatures below 250° F., although some modification of high temperature properties can also be expected. For example, brittleness induced at 885° F. has an effect on toughness at elevated temperature, according to Riedrich and Loib[103] and the return to original condition could only be achieved in their tests by heating the specimen to 1300 or 1500° F. The 885° F. embrittlement (detailed discussion of which is given in another section) would seem to be related to the sigma phase precipitation, but there is no unanimity of opinion on this point at the present writing. In any event, it seems probable that sigma phase will affect the physical properties of 27% chromium-iron alloy both at ordinary and elevated temperatures. Further investigation of this effect on the properties of the alloy is needed.

A fairly detailed discussion of the structural features of the 27% chromium-iron alloy has been presented, since its use for catalytic dehydrogenation processes will involve cyclic heating in the range from about 850 to 1350° F. This operating temperature includes the 885° embrittlement range and also the higher temperature range wherein sigma phase forms. The changes in structure and properties produced by long heating at these temperatures are, therefore, of paramount interest as regards the behavior of the alloy during service. Heretofore, the alloy has been used either under various chemical environments at temperatures below about 400° F., or as a high heat resisting alloy for service between 1500 and 2000° F. Sigma phase has not been encountered in these services, as the temperatures involved were either too low or too high for its formation. Its mode of occurrence and its effect on properties, therefore, has been largely of academic interest up to this time. The 885° F. embrittlement has been recognized by the reduced ductility encountered when the alloy is slowly cooled from higher temperature. Quick cooling through this range, as suggested by Becket and MacQuigg,[7] has long been practiced in annealing the alloy during manufacture in order to place it in the most ductile condition prior to service.

Embrittlement Phenomena

Sigma Phase Embrittlement has been already described in some detail in the foregoing section on structural characteristics. Most of the investigations that have been made along these lines were concerned mainly with fixing the limits of composition of sigma phase with a view of amending the constitution diagram rather than to determine the specific influence of sigma on the properties of high chromium-iron alloys.

Service data[143] indicate that transformation of a portion of the structure of these alloys to sigma is accompanied by an increase in hardness and by a loss in ductility. These effects become pronounced upon heating the metal for extended periods of time in the range of 1025 to 1300° F. Changes in mechanical properties depend on the amount of sigma precipitated — which, in turn, is a function of the composition of the metal and the service temperature conditions and exposure time.

On the basis of results obtained by The Babcock & Wilcox Tube Co.,[141] it may be stated that the best combination of initial physical properties in low carbon, wrought 27% chromium-iron alloys would be obtained by quenching the alloy from the 1400 to 1600° F. temperature range.

885° F. Embrittlement — Prolonged heating within the range of 700 to 1000° F., or slow cooling through that range, causes what is generally referred to as "475° C. brittleness" in 25 to 30% chromium-iron alloys. This phenomenon has not yet been satisfactorily explained, but some metallurgists think it is similar to the temper brittleness which occurs in some engineering alloy steels upon slow cooling after tempering. X-ray diffraction studies at The Babcock & Wilcox Tube Co.[134] indicate that this 885° F. brittleness may be associated with sigma phase and, therefore, it represents an incipient and very deleterious form of practically sub-microscopic grain boundary precipitation of the intermetallic compound FeCr. In

any event some atomic disturbance occurs, as is indicated by X-ray diffraction studies. Corrosion resistance in acid solution appears to be lowered when the metal is embrittled, but no data are available to indicate whether or not resistance to corrosion at elevated temperatures is affected. Average rates of corrosion in boiling 65% nitric acid for 27% chromium-iron alloy after aging at 885° F. (and after sigma phase has precipitated) may be compared with the rate for annealed metal by reference to the following data:[138]

Condition	Penetration Per Month
Annealed	0.000744 in.
Embrittled 500 hr. at 885° F.	0.002580
Embrittled 6000 hr. at 885° F.	0.009090
Sigma precipitated in 2900-hr. service	0.002080

The 885° F. embrittlement occurs in practically carbon-free alloys as well as in those of moderate carbon content. It also occurs in alloys to which nickel, silicon, vanadium, molybdenum, columbium or titanium has been added. While no corrective measure to avoid this brittleness is known, Franks[102] states that small nickel additions tend to slow down its development. The object of quenching after heating is to cool rapidly through the embrittlement range, which, as developed by Becket and MacQuigg,[7] is from 1100 to 750° F. Maximum softness is obtained by heating to a rather high temperature — usually between 1250 and 1650° F. — and rapidly cooling.

In a series of commercial iron-chromium alloys containing from 20.9 to 58.3% chromium, Becket[68] found that the hardness peak on heating occurred at 885° F. Holding time at temperature in his tests was 60 hr. This coincides with the peak hardness temperature determined in certain chromium-iron alloys by Riedrich and Loib,[103] and with the hardness-temperature curves for the 27% chromium-iron alloy determined in the laboratory of The Babcock & Wilcox Tube Co.[136] In the last mentioned tests, a temperature gradient bar was heated 400, 1000 and 5000 hr. and hardness was measured at the thermocouple locations along the bar length. The results of these tests are shown plotted in Fig. 16.

The time required for maximum embrittlement seems to vary in certain steels but, in general, considerable embrittlement may occur during short heating in the damaging temperature range without concomitant increase in hardness. Continued heating thereafter causes marked increase in hardness. Riedrich and Loib[103] found that a steel containing 29.3% chromium embrittled markedly in 60 min. at 885° F. and was "completely" embrittled in 240 min., although hardness only increased from 185 to Brinell 200. The effect of time at 885° F. on the hardness of a series of chromium steels is shown in Fig. 17. These data represent hardness values determined on three heats of 27% chromium-iron alloy at The Babcock & Wilcox Tube Co.[137]

Bagsar[133] reports that heating 30 min. at 1100° F. restored ductility in an embrittled 27% chromium-iron pipe which had been operated at 950° F. and had hardened to Brinell 250 to 260. Hardness dropped to 131 to 134, and bending properties were satisfactory with this treatment. Heating 5 to 7½ hr. at 975° F. caused embrittlement of thin chromium-iron sheet. In ⅝-in. plate, embrittlement occurred in 3 hr. at 925° F., as indicated by a drop from 195 to 20 ft-lb. in unnotched Charpy impact specimens. Conversely, heating the embrittled metal for 3 hr. at 1050 and 1100° F. restored the original impact strength of 195 ft-lb. in unnotched specimens.

In Becket's tests,[68] the necessity of higher and higher temperature to eliminate 885° F. brittleness as the chromium content of the metal increases was shown

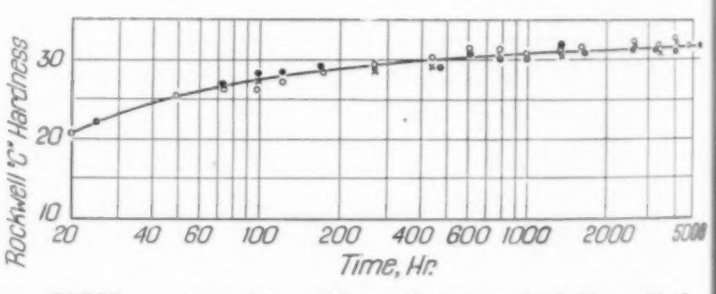


Fig. 17 — Effect of Time at 885° F. on Hardness of Three Heats of 27% Chromium-Iron[136]. Analysis range is same as given under Fig. 16

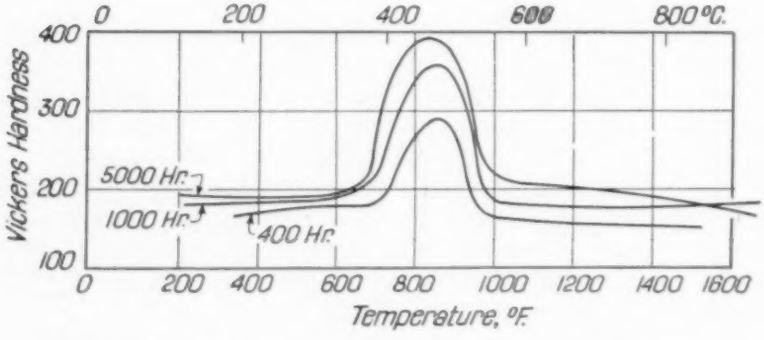


Fig. 16 — Hardness Surveys on Bars After Prolonged Heating in a Temperature Gradient[136]. Analysis: 0.20% max. C, 1.50% max. Mn, 0.025% max. S and P, 0.75% max. Si, 26 to 30% Cr, 1.00% max. Ni, 0.12 to 0.25% N_2

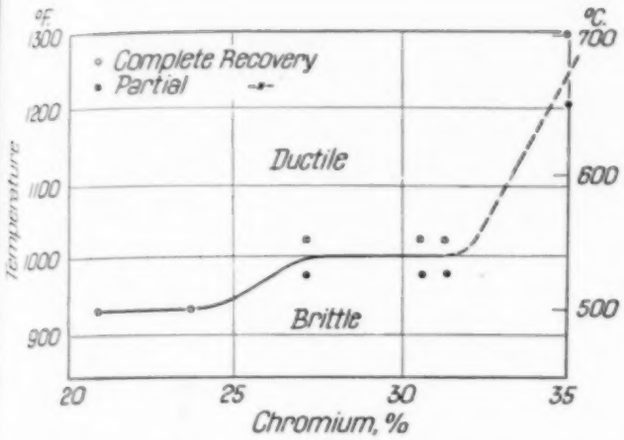


Fig. 18 — Effect of Temperature on Restoration of Ductility in Iron-Chromium Alloys Previously Embrittled by Heating at 885° F. (Becket[68])

for a series of commercial iron-chromium alloys (see Fig. 18). The alloys tested contained from 0.06 to 0.16% carbon, 0.37 to 0.57% silicon and 0.52 to 0.78% manganese.

The effect of aging time at 885° F. on the room temperature, static tensile properties of 27% chromium-iron alloy is illustrated in Fig. 19. From these graphs it will be observed that both yield and tensile strength increase while ductility decreases with longer aging time.

Properties may be pronouncedly impaired by 885° F. embrittlement before any considerable increase in hardness takes place. Nevertheless, prolonged sojourn of the alloy in the embrittlement range accompanied by marked increase in hardness, impairs the mechanical properties to the maximum extent. The susceptibility toward brittleness of 27% chromium-iron has been proven by varying the cooling rate during heat treatment.

Brittleness may be alleviated by rapid cooling from above 1100° F. Heating at 300 to 500° F. will improve toughness and will lessen the tendency toward breakage on deformation or from shock. A series of carefully controlled laboratory tests performed by The Babcock & Wilcox Tube Co. show that the alloy, when embrittled at 885° F. may have its toughness restored at temperatures of 1000° F. and higher, if heated for a long enough time. One hour suffices to remove it at 1100° F. or higher, 5 hr. are required at 1050, and 1000 hr. at 1000° F. These data are given in Table X on page 994.

Recent Embrittlement Studies

Aside from the data already given, additional information on the behavior of the alloy on aging has been developed by The Babcock & Wilcox Tube Co. in the course of the metals research program for Rubber Reserve Co. The titles of the reports covering these studies are listed in the bibliography (pages 1024 and 1028). The more important metallurgical features developed during these rather extensive investigations are given in the following paragraphs.

In the heating of gradient bars the peak embrittlement temperature was found to be 885° F., which corresponds to that usually ascribed to the alloy. Grain boundary widening occurs as has been described by Riedrich and Loib;[103] a typical example of this structural feature is shown in Fig. 20, page 995. "Overaging", even on heating as long as 13,000 hr., does not occur at the peak embrittlement temperature but the widened grain boundary effect seems to disappear when the embrittled metal is heated in the curative range of temperatures — that is, 1050° F. or higher. Gross sigma precipitate may occur in annealed metal at a temperature as low as 985° F. on extended heating; slightly higher temperatures accelerate the precipitation. In the 885° F. embrittlement range, mechanical twins or Neumann bands may form

(Continued on bottom of page 993)

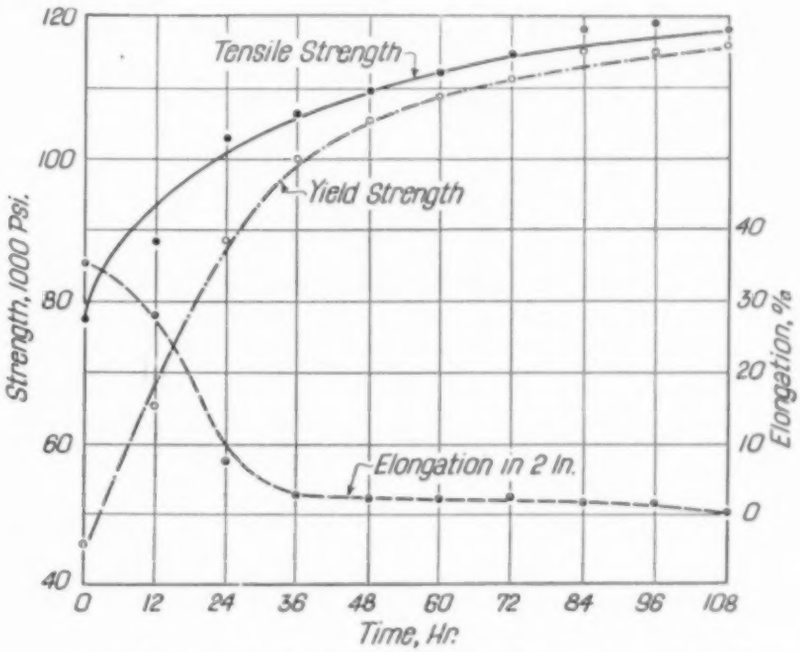


Fig. 19 — Graph Showing the Effect of Aging Time at 885° F. on the Room Temperature Tensile Properties of 27% Chromium-Iron Alloy. (The Babcock & Wilcox Tube Co.[137])

International Control of Atomic Energy

ON March 16, the State Department issued a 76-page booklet* entitled "A Report on the International Control of Atomic Energy", with the implied approval of the committee appointed by the Secretary of State to study the safeguards necessary to protect the United States up to the time a United Nations' commission, as proposed by the President and Premiers of the United States, Canada and England, can take over the control of atomic energy and other weapons of mass destruction. This committee consisted of Dean Acheson, Under-Secretary of State and chairman, Vannevar Bush, chairman of National Defense Research Council, James B. Conant, president of Harvard University, Gen. Leslie R. Groves, head of Manhattan Engineer District, and John J. McCloy, formerly assistant Secretary of War.

In order to formulate a definite plan, this committee in turn appointed the following board of consultants: David E. Lilienthal, chairman of Tennessee Valley Authority, Chester I. Barnard, president of New Jersey Bell Telephone Co., J. Robert Oppenheimer of California Institute of Technology, Charles Allen Thomas, vice-president and technical director, Monsanto Chemical Co., and Harry A. Winne, vice-president in charge of engineering policy, General Electric Co.

The booklet mentioned above is the result of seven weeks' constant study of this board of consultants. "Five men of widely differing backgrounds and experiences who were far apart at the outset found themselves, at the end of a month's absorption in this problem, not only in complete agreement that a plan could be devised but also in agreement on the essentials of a plan." This general plan will now be outlined briefly, the quotes being words used by the board of consultants.

"WE HAVE CONCLUDED unanimously that there is no prospect of security against atomic warfare in a system of international agreements to outlaw such weapons controlled *only* by a system which relies on inspection and similar police-like methods" because national rivalries in the construction of weapons "places so great a pressure upon a system of international enforcement by police methods that no degree of ingenuity or technical competence could possibly hope to cope with them". Any such system of inspection "will inevitably be slow to take into account changes in the science and technology of the field; the activity would offer the inspectors a motive pathetically inadequate to their immense and dreary task"; sufficient men of proper intelligence could not be recruited; and such intensive control and inspection as would be necessary "involves a persistent challenge of the good faith of the nations inspected, tending to produce internal as well as external political problems".

This dilemma is avoided by recognizing that there are certain activities that are intrinsically "dangerous" — inimical to the peace of the world — for any nation or its citizens to carry on — namely *(a)* the mining, refining and ownership of the raw materials uranium and thorium, *(b)* the production, separation and ownership of the fissionable materials plutonium and U^{235} and *(c)* the use of fissionable materials for the making of weapons. These dangerous activities should be the exclusive prerogative of an international agency subscribed to by all nations — the "Atomic Development Authority". On the other hand, "safe" activities that may be carried on by nations or their citizens under license and reasonable inspection are the use of radioactive material on loan as tracers in scientific, medical and technical study, the use on loan of small quantities of denatured U^{235} or plutonium for neutron sources or gamma ray production, and the use of larger quantities loaned the Atomic Development Authority — but still a fraction of the amount needed for an atomic bomb — for the production of power in reactors generating up to 1,000,000 kw. per unit.

These proposals are based on the fact that "the only scientific evidence worthy of regard makes it clear that in terms of security uranium is indispensable in the production of fissionable material on a scale large enough to make explosives or power. Uranium is the only natural substance that can maintain a chain reaction. It is the key to all foreseeable applications of atomic energy." While thorium of itself cannot maintain a chain reaction, "with a fairly substantial amount of uranium to begin with and suitably large quantities of thorium, a chain reaction can be established to manufacture material which is an atomic explosive and which can also be used for the maintenance of other chain reactions".

Of greatest importance is the circumstance that fissionable U^{235} and plutonium can be denatured in such a way as to render them harmless as explosives yet unimpaired for the "safe" applications of research, radiators, and power sources. To remove the denaturant would require a large and complex installation, a high degree of scientific and engineering skill, and considerable time.

ONCE the Atomic Development Authority is organized, accepted, and financed, its first task "will be to bring under its complete control world supplies of uranium and thorium; then to conduct continuous surveys so that new deposits will be found and so that the agency will have the most complete knowledge of the world geology of these materials. It is apparently the view of the authorities that these elements occur in high concentrations only under very special geologic conditions. This would seem to mean that the areas which need to be surveyed, to which access must be had, and

*Obtainable for 20¢ from the Supt. of Documents, U. S. Government Printing Office, Washington 25.

which would ultimately have to be brought under control, are relatively limited.

"If the Atomic Development Authority is the only agency which may lawfully operate in the raw materials field, then any visible operation by others will constitute a danger signal. This situation contrasts vividly with the conditions that would exist if nations agreed to conduct mining operations solely for proper purposes; for surreptitious abuse of such an agreement would be very difficult to detect. It is far easier to discover an operation that should not be going on at all than to determine whether a lawful operation is being conducted in an unlawful manner . . . . .

"The second major function of the Authority would be the construction and operation of useful types of atomic reactors and separation plants. This means that operations, like those at Hanford and Oak Ridge and their extensions and improvements, would be owned and conducted by the Authority. These production plants are intrinsically 'dangerous' operations. Indeed they may be regarded as the 'most dangerous', for it is through such operations that materials can be produced which are suitable for atomic explosives."

Third, the Atomic Development Authority will vigorously conduct research in all fields of atomic energy, "for only by preserving its position as the best informed agency will its staff be able to tell where the line between the intrinsically dangerous and the non-dangerous should be drawn. If it turns out at some time in the future, as a result of new discoveries, that other materials than now known lend themselves to dangerous atomic developments, it is important that the Authority should be the first to know this fact. At that time measures would have to be taken to extend the boundaries of safeguards."

Finally, the Authority would lease denatured fissionable materials for approved purposes wherever "the Authority could have access to the establishment in which such material is used". The board of consultants believed that the Authority would be able to design power piles that are not readily convertible to any "dangerous" operation as soon as enough fissionable material could be produced for the operation of such power piles. It is desirable that this planning stage be unified with requirements for inspection, for the "operations can themselves be so conducted as to make this inspection and control easy".

Such activities of geologists, chemists, physicists, engineers, auditors and administrators, it is believed, would attract to the Authority personnel from all nations of the highest quality, for they would be working in a field of knowledge with enormous prospects for future development.

WHEN the mining or refining of uranium and thorium by individuals or nations is illegal and is the sole prerogative of the Atomic Development Authority, as well as the construction of reactors or power piles, a calculated attempt at evasion may still be imagined. "But a surreptitious program of atomic armament on a scale sufficient to make it a threat is so vast, and the number of separate difficult undertakings so hard to conceal, that this effort should be impossible to hide. The fact that it is the existence of the effort rather than a specific purpose or motive or plan which constitutes an evasion and an unmistakable danger signal is to our minds one of the great advantages of the proposals we have outlined."

The Secretary of State's committee was impressed by the fact that this plan recommended by its board of consultants preserved "the security of the United States both during the period of an international discussion and during the period required to put the plan into full effect". Disclosure of our scientific and technical information, relinquishment of our stockpiles of uranium, transfer of title to our reduction plants and refineries would be step by step, and our present superiority in the field of atomic weapons would only be relinquished when the presence of the full-functioning Atomic Development Authority would provide us with a truer measure of safety than America could achieve through her own efforts, alone.

due to strain from volume change, as shown in Fig. 21, page 995.

Sigma phase has not been detected by X-ray diffraction after heating at 885° F. but an atomic disturbance occurs as is indicated by the diffused rays in the back reflection photograms shown in Fig. 22, page 996.

Phase changes are stimulated by cold working the metal. Worked metal given an 80% reduction of section shows characteristic X-ray spectra for sigma precipitate when aged 1000 hr. at 950° F., and also at temperatures between 1000 and 1200° F. Typical diffraction patterns for cold worked chromium ferrite aged 1000 hr. at 885° F. and for metal cold worked and aged at 1000° for 1000 hr. are shown in Fig. 23, page 996. The characteristic line spectrum of sigma is present in the latter example while the former has a pattern similar to annealed chromium ferrite. These changes are also discernible on microscopic examination. In spite of its initially hardened condition, the cold worked metal ages substantially and increases in Rockwell hardness from C-23 to C-39. Typical microstructures of a cold worked and aged series of samples are shown in the data sheet, page 976-B. Once formed, sigma precipitate is relatively stable and requires a temperature of 1500° F. or higher for its resolution in a practicable time. A series of heating tests show that greatly extended time is required at 1400° F. for going

into solution, while at 1300° F. the sigma precipitate will not transform back to ferrite.

Working and Fabricating

Forging — The high chromium, low carbon stainless iron alloys may be forged and hot worked without undue difficulty, provided a proper sequence of casting, reheating and working is followed. The A.S.T.M. Tables of Data[113] makes the following recommendations for the forging of Type 446 stainless steel:

Initial forging temp.	1900 to 2000° F.
Final forging temp.	1300 to 1450° F.
Method of cooling	Air

Some investigators[6, 130] recommend an initial temperature as high as 2100° F. United States Steel Corp. and others[74] recommend preheating at 1400 to 1500° F. followed by an initial forging temperature of 1900 to 2000° F., finishing to 1450° F. and air cooling from the hammer.

The following practice for forging ingots has been followed, with satisfactory results, by The Babcock & Wilcox Tube Co.: *(a)* Remove from molds 1½ hr. after pouring. *(b)* Place in soaking pit held at 1500 to 1600° F. *(c)* Hold until uniform temperature is obtained. *(d)* Heat 75° F. per hr. to 1900° F. *(e)* Hold 5 hr. *(f)* Forge. Forging at this plant is done under a 16,000-lb. hammer and blooms are finished at about 1550° F. Increasing the initial forging temperature above 1900° F. has resulted in cracked blooms. After forging, the blooms may be air cooled for conditioning and then are reheated and rolled into round bars.

The temperature ranges cited for forging may be used also for other hot working operations such as turning Van Stone laps, upsetting and hot swaging. The minimum temperature found to produce satisfactory metal flow should be used because of its beneficial effect on the microstructure of the metal.

Forging should not be conducted at too low a temperature, however, if internal rupture is to be avoided.

Hot Rolling — These alloys may be hot rolled into bars, rods, flat products (plate, sheet and strip), and tubing. Lateral spread on rolling being greater for high chromium alloys than for ordinary steel,[131] proper allowance should be made in the shape of the rolling passes.

According to Johnson and Sergeson,[51] not all the stainless types may be rolled direct from the ingot. The complex alloys and 25 to 30% chromium-irons must be forged into a billet. Rolling and forging ranges are narrow; pyrometric control is a necessity. The lower chromium steels are easier to work, offering less resistance to flow. Ingots are charged hot into soaking pits with fires off in order to avoid rupturing and save time. The

Table X — Effect of Time and Temperature in Removing 885° F. Embrittlement in 27% Chromium-Iron*
The Babcock & Wilcox Tube Co.[140]

Hours at Heat	Property†	Temperature of Corrective Heat				
		950° F.	1000° F.	1050° F.	1100° F.	1200° F.
1	Yield	125,500	116,200	120,700	55,000	52,000
	Ultimate	127,000	116,200	120,700	80,000	81,000
	Elongation	0.0	0.0	0.0	34.0	34.0
	Hardness	332	341	347	177	175
5	Yield	117,500	109,000	51,500	56,500	—
	Ultimate	122,000	114,000	81,000	82,000	—
	Elongation	1.0	1.0	34.0	33.0	—
	Hardness	336	313	175	181	—
10	Yield	110,500	102,700	53,300	57,500	52,000
	Ultimate	120,000	113,400	81,500	80,500	81,500
	Elongation	1.5	13.0	33.0	33.0	33.5
	Hardness	318	283	178	177	175
25	Yield	107,500	103,200	55,000	51,500	57,000
	Ultimate	114,000	111,000	81,250	80,000	81,000
	Elongation	1.5	3.0	33.0	34.0	33.0
	Hardness	310	270	175	175	178
50	Yield	111,000	107,800	59,000	54,000	57,000
	Ultimate	119,000	107,800	81,000	81,800	79,500
	Elongation	3.5	3.0	33.5	31.5	33.0
	Hardness	314	290	177	178	174
75	Yield	110,000	107,000	51,500	51,000	60,000
	Ultimate	114,000	112,500	81,000	79,000	80,000
	Elongation	1.25	5.5	34.0	34.0	33.0
	Hardness	308	297	180	175	176
100	Yield	104,000	109,500	53,000	51,000	56,000
	Ultimate	114,000	110,500	81,000	80,000	81,500
	Elongation	0.5	1.25	32.5	33.0	32.5
	Hardness	298	302	175	175	177
500	Yield	92,200	106,000	52,500	55,000	56,000
	Ultimate	113,000	114,000	81,150	83,000	80,500
	Elongation	—	4.0	33.0	30.0	31.0
	Hardness	—	303	179	181	176
1000	Yield	97,700	57,100	54,000	53,000	60,000
	Ultimate	103,500	82,250	82,900	80,000	82,000
	Elongation	1.5	29.5	31.0	30.0	26.0
	Hardness	291	211	187	176	175

*Analysis: 0.15% C, 0.80% Mn, 0.008% P, 0.014% S, 0.48% Si, 27.71% Cr, 0.18% Ni, 0.14% N_2.

†Condition of material at beginning of test (prior to heating but subsequent to embrittlement of 500 hr. at 885° F.) was as follows: Yield strength, 126,500 psi.; ultimate strength, 132,000 psi.; elongation in 2 in., 0.0%; Brinell hardness, 340.

chromium-iron series require more care in this respect than the chromium-nickel austenitic alloys. Since conductivity is low, extra heating time is necessary; too long a heating enlarges the grains on the surface and increases susceptibility to cracking. Decarburized surfaces will also cause trouble. After the ingot is reduced to slab or billet size the surface must be prepared by chipping and grinding to remove all defects preparatory to rolling into bars, rod and strip.

Johnson and Sergeson[40, 54] recommend slow speed hand mills for bars rather than continuous trains. They state it is impossible to roll stainless steels with the same setup used for carbon or alloy steels, since the stainless alloys tend to spread in the rolls, require tighter guides and tend to be galled by the guides which tear, sliver and scratch the rolled bars. Commercially important sizes can be produced by the hand round method, and smaller sizes by guide mills, properly operated. Sheets are rolled from ground slabs reheated in clean furnaces. Slabs are broken down on a jobbing mill using 15 to 25% reduction per pass; water under pressure keeps the surfaces clean and the roughing pass rolls cold. Plates are flattened while hot and sheared into sheet bar sizes while still warm. Sheet bar is pickled and conditioned for rerolling; starting and finishing temperatures for rolling sheets of 30% chromium alloy are 1500 to 1800° F. and 1200 to 1500° F. respectively.

MacQuigg[9, 36] states that high chromium steels roll or forge about as well as medium carbon steel provided carbon is below 0.50%. They are stiffer than carbon or low alloy steel so that more power is necessary to work them and the hot working temperatures must be more closely controlled. Initial temperatures as high as 2100° F. are used in forging and rolling ingots. MacQuigg recommended 1740 to 1925° F. as finishing temperatures (depending on the operation) with 1470 to 1560° F. as the lowest permissible range. He also remarked that trouble in hot working is more closely related to carbon than to chromium content.

Franks' investigations[41, 55] revealed that nitrogen improves rolling and forging properties; also, that 20 to 30% chromium steels containing less than about 0.25% carbon can be readily hot worked.[91] Recommended initial hot working temperatures are in the neighborhood of 2000° F. with finishing temperatures not much below 1400° F. Franks' statement[91] that ingots are generally broken down on a blooming mill is contrary to the forging recommended by Johnson and Sergeson.[54] Curtiss[54] mentions finish rolling from slabs to plate in one heating, this being different from the usual conditioning and reheating in rolling other flat products.

In producing bars for piercing into tubing, at the steel plant of The Babcock & Wilcox Tube Co., the 27% chromium alloy ingots are forged into blooms as already described; following this they are air cooled and surface conditioned preparatory to rolling into bars. Reheating of blooms is done in a pusher type furnace fired with fuel oil; initial rolling temperature is 2150° F., finishing about 1800° F. Rounds are rolled from 7 in. diameter to 3 in. diameter on a 24-in., three-high bar mill. When rolling rounds below 5 in., the 7⅜-in. blooms are stopped at 5⅜ in. and are cooled down, cut

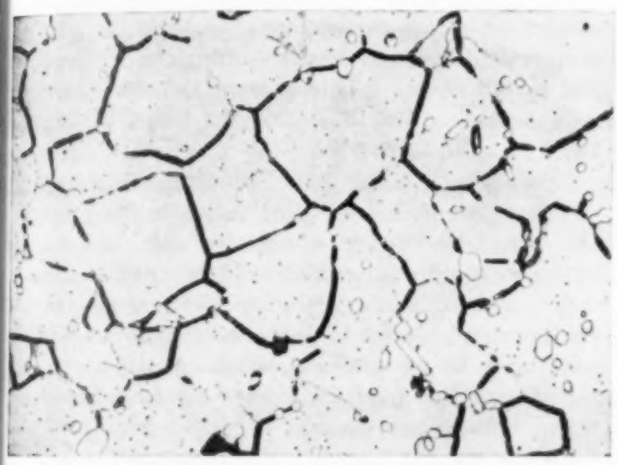


Fig. 20 — Grain Boundary Widening in 27% Chromium-Iron Alloy Heated in the 885° F. Embrittling Range[140]. Etched with aqua regia. 750×

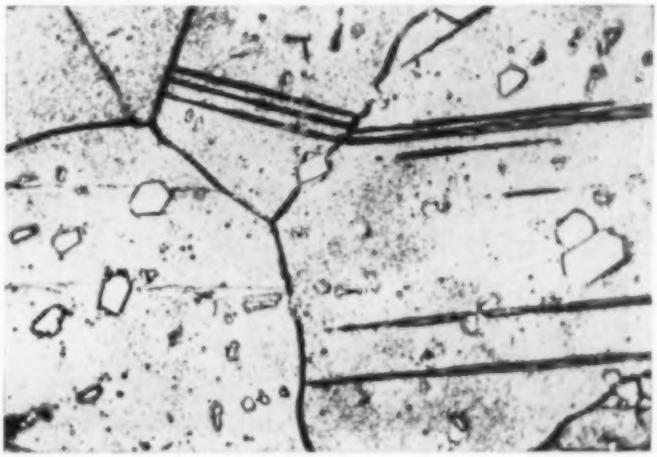
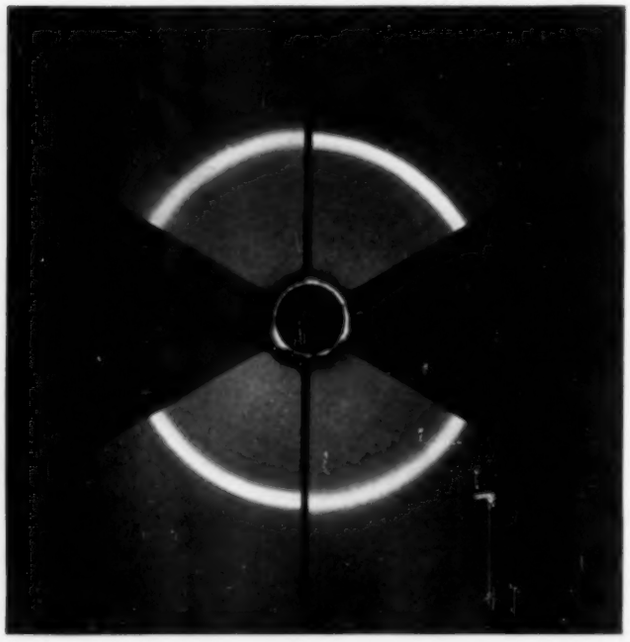


Fig. 21 — Neumann Bands or Mechanical Twins in Chromium Ferrite, Whose Mottled Appearance Suggests a Sub-Microscopic Precipitate[137]. Sample aged 6000 hr. at 885° F. Deep aqua regia etch. 500×

Fig. 22 — Back Reflection Photogram of an Embrittled and Annealed Sample of 27% Chromium-Iron. Upper left and lower right show diffuse rings after aging 13,000 hr. at 885° F. Compare with identical material aged and reannealed — lower left and upper right. (The Babcock & Wilcox Tube Co.[134])



in halves and reheated in order to get rounds of good surface and dimensions. Round bars are finished oversize and are prepared for piercing by rough turning to remove all surface defects.

Piercing — The present writer has described in another paper[54] the piercing and rolling of stainless steel tubes, including the ferritic chromium ones containing 16 to 18% and 25 to 30% chromium. Being relatively soft when hot, these alloys are easily marked by guides or rolls; overheating causes grain growth and results in poor or ruptured surfaces. Piercing the chromium-irons does not require much, if any, more power than piercing carbon steels. Temperature control should be strictly maintained during piercing, rolling and finishing. Piercing is usually done at 2075 to 2125° F.; rolling, reeling and finishing at lower temperatures. Finishing temperatures should be controlled to obtain a suitable structure for further working by cold drawing.

Ihrig[119] has tested the ferritic stainless steels by his "hot twist" test and reports that maximum hot ductility is obtained for Type 446 stainless at 2350° F., which is higher than is normally employed for piercing this alloy. Data by the present author, not previously published, show a somewhat different result for 27% chromium-iron alloy; test pieces twisted a greater number of turns at lower temperature (see Fig. 24). These tests were conducted on $\frac{9}{16}$-in. test rounds, held 30 min. at temperature and twisted to failure at a speed of 130 to 140 r.p.m. The test bars had the following composition: 0.15% C, 0.48% Mn, 0.35% Si, 26.84% Cr, 0.50% Ni, 0.136% N_2.

Bending, Coiling and Spinning — Light gages of 27% chromium-iron alloy, when in the properly annealed and ductile condition, may be bent at ordinary temperature. However, notches or surface marks and sudden application of stress may result in fracture. Moderate preheating to about 300° F. will tend to maintain a tough condition in the metal and is good insurance against breakage.

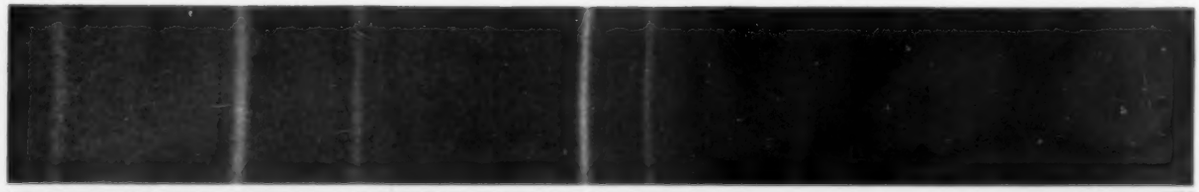


Fig. 23 — Typical Diffraction Patterns of Cold Worked Chromium Ferrite. Top — After aging 1000 hr. at 885° F. the lines for ferrite are sharp. Bottom — after aging 1000 hr. at 1000° F. many additional lines corresponding to sigma phase appear. (The Babcock & Wilcox Tube Co.[134])

Long radius bends are best made by such practice although it will often be found that thin gage tubes can be bent cold. Bends of short radii (less than six times the diameter) are best made hot — that is, between 1200 and 1400° F.

Coiling should be done between 1500 and 1200° F. Tubes, especially the larger

sizes, should be sand filled prior to coiling to maintain good form. Temperature prior to coiling may approach 1800° F. but the work is preferably started at the lower temperature.

According to Curtiss,[54] chromium-iron alloy plates, 1¼ in. or thicker, should never be rolled for bending in a chilled or cold condition; they should be warmed up and rolled slowly. Ends of heavy plates should be pressed to proper curvature before rolling. All except light material should be heated to about 1500° F. for bending or rolling. For forming by pressing, the straight chromium-iron should be heated to 1850° F. maximum and all work done below 1500° F.

Spinning of tube ends may be done in the usual manner using hardened toolsteel, alloy iron, Stellited or other hard-faced tools. Tools of roll or bar form may be used and tube ends should be smoothed, after cutting, to eliminate notches. The tubing should be preheated to 300 to 500° F. before applying tool pressure so as to avoid breakage. Lubricants such as heavy graphited grease are desirable to prevent pickup and galling.

Allegheny Ludlum Steel Corp.[67] states that 28% chromium-iron alloy, because of its properties, is not very adaptable to deep drawing or forming operations. It may be drawn or formed into simple shapes requiring shallow draws, and bends can be made from it. Heating to 300 to 400° F. will aid in the bending operations. Likewise, the alloy may be spun into simple shapes but does not possess the ductility required for complicated forms.

Shearing, Punching and Riveting — Hostettler[54] points out that the chromium steels break when cut partly through (50 to 75% of their cross-section), shearing like carbon steel in this respect. Shearing of equivalent areas requires more power than for plain steel; good sharp blades should be used. The high chromium-iron may break out at the edges of the cut and, if so, the plate or sheet in that heat should be preheated to about 300° F. In some cases, the material may be sheared and cold punched without difficulty. Welded specimens, however, crack severely at the weld and fusion zone on cold shearing. Warming to 400 to 600° F. improves fabricating properties to a marked extent.

United States Steel Corp.[80] states that straight chromium stainless steels may be blanked and punched with dies and punches set as for carbon steel. More power is required than for carbon steel and slower speed is advisable. It is imperative to have and maintain very sharp punches and dies. Perforating may be done in like manner, keeping tools sharp and reducing the speed of the press to half or one-third that used for mild steel. Minimum diameter of perforations equals the thickness of the stock.

In riveting chromium ferrites, Mitchell[84] recommends ground rivets to remove all surface markings, since sharp re-entrant angles are dangerous. Rivets should have a $\frac{1}{16}$-in. radius fillet between head and shank and should be driven into chamfered holes. Hydraulic riveting is recommended by McEvoy[84] for stainless iron; rivets should be driven hot — that is, at 1450° F., more or less. Caulking should be carefully done to avoid flaking at the edges; indenting or scoring of the under plate must be guarded against. Allegheny Ludlum Steel Corp.[67] states that on account of its low ductility when cold, 28% chromium alloy should be hot riveted and temperatures should not exceed 1500° F. Rivets that exhibit surface defects or have been overheated should be discarded. Rivet holes can be drilled or punched; the former method is recommended but if the holes are punched they should then be reamed. Standard Oil Co. of Calif.* recommends that heavy peening, bobbing or riveting should be done either in the 400 to 800° F. range or the 1200 to 1600° F. range. Cold riveting with 18-8 or 25-20 rivets can be accomplished if care is exercised to avoid pounding the 27% chromium steel sheet.

Machining and Sawing — Colbeck and Garner[77] found that nitrogen improves machinability of low carbon, high chromium steels. MacQuigg[91] observes that machinability depends more on structure than on chromium content and, if annealed properly, a steel containing 25 to 30% chromium and 2 to 2.75% carbon can be machined. According to Franks,[91] the hardness of carbides present in 25 to 30% chromium steels makes these alloys more difficult to machine than ordinary low carbon steels.

Palmer[54] states that the high frictional characteristics of the stainless steels persist in the ferritic high chromium alloys, and

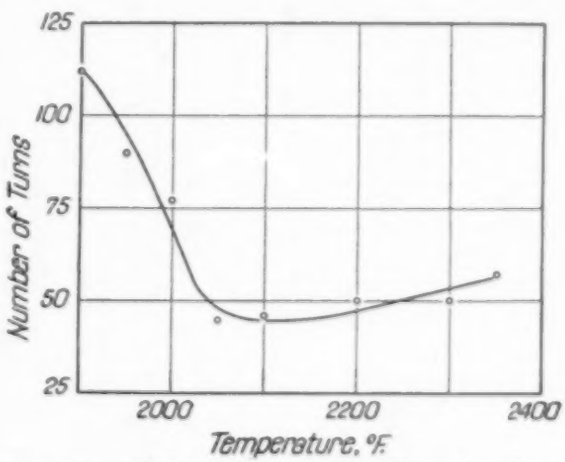


Fig. 24 — Twist Tests on 27% Chromium-Iron Alloy. (The Babcock & Wilcox Tube Co.[133])

*See Section H, p. 8 of reference No. 133.

Table XI — Physical Properties of Arc Welded 27% Chromium-Iron Alloy, Tested at 70° F.

Source	Chemical Analysis: Base Metal Cr	Base Metal N_2	Electrode C	Electrode Cr	Electrode Ni	Electrode N_2	Preheat Temp.	Heat Treatment of Welded Piece	Yield Point	Tensile Strength	Elongation in 1 In.	Red. of Area	Bend Angle	Impact Value Ft-Lb. Notched	Impact Value Ft-Lb. Not Notched
Eberle[127]	27			27	4		300° F.	None	59,870	89,730	13.2*(b)*	35.1	180°*(a)*	12, 16	56, 86
				25	20									17.5	187, 220
				27	4		300	500 hr. at 1300° F.						10, 12.5	76.5, 330
				25	20									14.0	
				27	4		300	*(c)*						15.5, 18.5	
				25	20									23.0	
				27	4		300	500 hr. at 885° F.						8.0, 8.5	
				25	20									13.0	
Thum[54]	26.0	*(d)*	0.25	28.76	0.56	0.10	Not stated	Presumably none		53,000	Nil	Nil	Nil	1.0	4.0
	26.0	*(d)*	0.21	26.95	0.21	0.33		Presumably none		56,000	Nil	Nil	Nil	2.5	18.0
Mueller, Carlson and Seabloom[118]	27.0	0.15	Type 446 (28 Cr)				None	½ hr. at 1450° F.*(e)*	60,400	78,800	22		20*(f)*	3.25*(g)*	
			Type 446 (28 Cr)				450	½ hr. at 1450° F.*(e)*	57,200	75,900	14.5		24*(f)*	0.75*(g)*	
			Type 329 (25-3 Cr-Mo)				None	½ hr. at 1450° F.*(e)*	64,000	81,000	23.0		20*(f)*	5.25*(g)*	
			Type 329 (25-3 Cr-Mo)				550	½ hr. at 1450° F.*(e)*	60,300	74,200	22.0		24*(f)*	5.00*(g)*	
			Type 310 (25-20)				None	½ hr. at 1450° F.*(e)*	59,800	76,900	13.5		22*(f)*	9.00*(g)*	
			Type 310 (25-20)				450	½ hr. at 1450° F.*(e)*	51,500	75,800	23.0		28*(f)*	12.5*(g)*	
			Type 309 (25-12)				None	½ hr. at 1450° F.*(e)*	44,300	73,100	29.5		24*(f)*	12.5*(g)*	
			Type 309 (25-12)				None	None	56,200	80,300	24.0		20*(f)*	15.4*(g)*	
Franks[55]	25.0	0.24	Same as base metal*(h)*				Not stated	None	66,500	92,700	3				
								4 hr. at 1605° F.*(e)*	57,100	80,500	7				
			Same as base metal				Not stated	None	64,000	85,500	7				
								4 hr. at 1605° F.*(e)*	53,600	82,400	10				

(a) Guided jig bend tests with root of weld in tension.
(b) Elongation in 2 in.
(c) 500 hr. at 1300° F. and tested at 300° F.
(d) Carbon 0.10%.
(e) Air cooled after heating.
(f) Face bend tests; % elongation in ¼-in. circle.
(g) Substandard Charpy specimens (0.394x0.147 in.).
(h) Oxy-acetylene weld; all others arc welds.

when carbon is over 0.20% (as it frequently is in castings) hardness may also become a factor in machining. Extensive machining is not usually required with these alloys, but they can be turned at 50 to 70% of screw-stock speed, and can be threaded or tapped at slower speed. Cold drawn bars are probably more machinable than hot rolled bars. For sawing, the methods used for 0.30 to 0.40% carbon steel are satisfactory.[74]

Rustless Iron & Steel Corp.[71] suggests sulphur base oils, either straight or diluted with paraffin oil, for all machining operations on stainless steels. Carbide tooling is not generally recommended but has been used with some success in certain operations. High speed or cobalt steel tools are essential, the cobalt alloy steel being used on heavy uninterrupted turning. Allegheny Ludlum Steel Corp.[67] states that the 28% chromium grade tends to gall; therefore, tools of a design for half-hard brass are preferred. Drilling should be done with high speed drills at approximately the following speeds and feeds:

Diameter	R.P.M.	Feed
⅛ in.	1200	0.004 in.
¼	600	0.006
½	300	0.010
1	160	0.010

Clearance angle on the drill can be increased to 12 to 15° for ferritic type alloys.

According to one writer,[86] the ferritic stainless steels are easier to machine than those of austenitic structure. Reduced speeds and heavy feeds are the rule, using 40 to 60 linear ft. per min. when turning heavy work. Taps should have ground threads as for soft brass, but with slightly more taper on two or three full teeth. In threading,

a four or five-thread lead, instead of the customary two or three-thread lead, should be used with lard oil and sulphur mixtures, or a suitable commercial compound, as the lubricant.

Welding the 27% chromium-iron alloys may be done by practically any of the fusion methods recommended for mild steels — metal arc, oxy-acetylene flame, atomic hydrogen. These alloys cannot be forge welded or hammer welded.

Although their thermal expansion is actually slightly less than that of mild steels, some method of clamping the work, and the use of chill bars is desirable, especially in fabricating light parts. Electrodes or filler rods of the same composition or of slightly higher chromium content than the parent metal being welded should be used. Where greater ductility in the weld metal is necessary, it is sometimes possible to use electrodes of austenitic composition such as 18-8 Cr-Ni or 25-12 Cr-Ni or preferably 25-20 Cr-Ni. A heat treatment satisfactory for most welds consists of heating the weldment at 1400 to 1600° F. for 1 hr. per in. of thickness, and then cool rapidly. Stress relieving after welding is performed at slightly lower temperature (in the neighborhood of 1375° F.) to avoid distortion.

The mechanical properties of welded 27% chromium-iron alloys have been experimentally determined by several investigators. Typical results obtained in some of these investigations are summarized in Table XI. From these and other data,[54] it appears that nitrogen-bearing electrodes enhance the mechanical properties of welds made in 27% chromium-iron alloys. Such electrodes may be used where it is desirable to weld the alloy with the same composition as the base metal and, for chemical reasons, to exclude nickel from the deposited metal. Otherwise it would seem preferable, where maximum toughness is desired, to weld with 25-20 chromium-nickel rod or 27-4 chromium-nickel electrode.

Pickling, Cleaning, Passivating — For removing scale, after hot working or annealing operations, any one of several pickling solutions may be used following usual practice for stainless steels. The 25 to 30% chromium-irons do not pickle readily, and it may be necessary to rot the scale in one solution, scrub the surface to mechanically detach the sludge, and then follow by finish pickling and passivating.

For rough pickling of the heavier scale, the following may be used: 10 to 15% sulphuric acid (50° Baumé) plus 5 to 25% commercial hydrochloric; mixture at 130 to 160° F. Finish pickling may be in hydrochloric acid (10 to 15% by weight at 140 to 150° F.) followed by mixed nitric (10%) and hydrofluoric acids (0.5%) at 130 to 140° F.

Passivation, to remove iron contamination and other foreign matter on the surface, may consist of 15 to 30 min. immersion in 20 to 30% nitric acid by volume at 130° F.

Sandblasting is quite frequently resorted to for cleaning the straight high chromium-iron alloys and will be found more satisfactory than pickling except where light scales are involved. The Babcock & Wilcox Tube Co. sandblasts tubing of the alloy, followed by a light pickle and nitric acid dip. Shotblasting with steel shot is not recommended for obvious reasons.

The newer methods of descaling in molten mixtures of caustic and sodium hydride (or other molten alkaline salts), followed by quenching and pickle dipping, provide effective methods of cleaning chromium-iron alloys from annealing scale.

Heat Treating (Annealing)

Low carbon, wrought 25 to 30% chromium-iron alloys is heat treated to improve its ductility after hot rolling operations, to recrystallize and soften cold worked metal, or to relieve stresses following welding. As no allotropic transformations are involved, the treatments are most appropriately termed "annealing".

Beckett and MacQuigg[6] patented a method of making chromium-iron sheets or articles ductile by quickly cooling them through the range of 1100 to 750° F.; this was to avoid the embrittlement resulting on slow cooling through this same range. Pierce and Grossmann[54] state that heat treatment is applied to the 25 to 30% chromium alloys to modify their mechanical properties, since corrosion resistance is not affected, and if the material is hard or lacks ductility due to rolling or forging operations it may be annealed by heating to 1450 to 1650° F. and cooling rapidly. Beckett[54] states that high chromium alloys may be softened and ductility increased by quenching from around 1650° F. — also, that cold worked articles recrystallize rapidly only after heating at 1830° F. Kinzel and Franks[91] recommend long heating between 1565 and 1650° F., followed by rapid cooling.

Colbeck and Garner[77] have shown that an excellent combination of properties results from water quenching the nitrogen-containing chromium steels from higher temperatures, namely, 1920 to 2200° F. These temperatures are higher than are employed in current American practice. Johnson and Sergeson[91] state that sheets of 25% chromium steel are heated to 1650° F. and cooled rapidly for annealing.

In current practice on 23 to 30% chromium alloys, Parmiter* recommends 1300 to 1350° F.

*Metals Handbook, 1939 edition, p. 539.

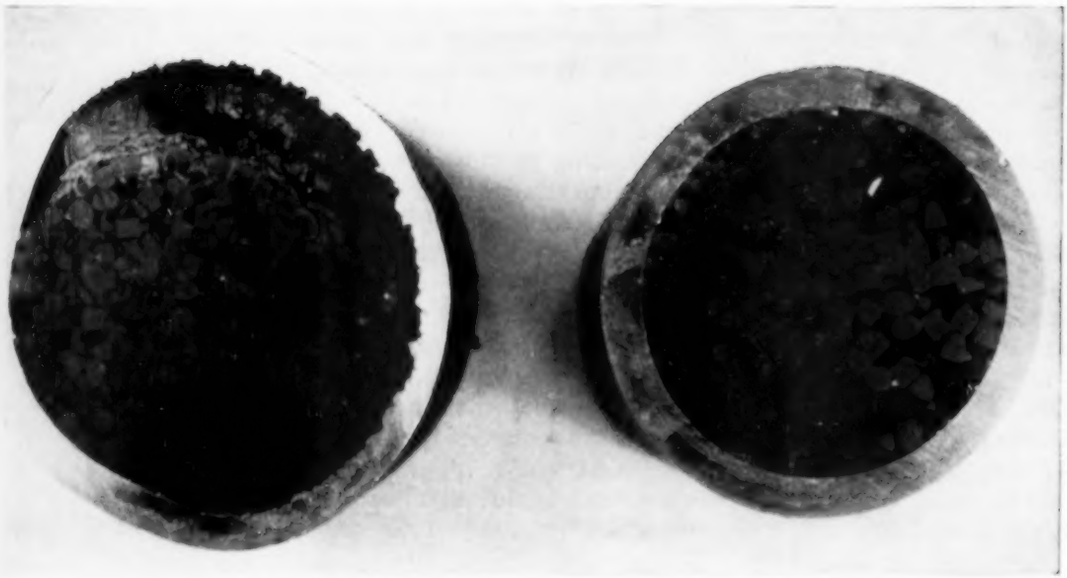


Fig. 25 — Sample Tubes Removed From Dehydrogenation Service, One Badly Overheated, Carburized and Bulged; Both Are Plugged With a Mass of Fused Catalyst and Coke[142]

with air cooling for softening, and 1550 to 1600° F. with water quenching for annealing.

Several authorities[71, 74, 113] recommend rapid cooling from 1450 to 1550° F. for full annealing the Type 446 stainless alloy. Allegheny Ludlum Steel Corp.[67] cautions that 1650° F. should not be exceeded in the annealing operation, and agrees with United States Steel Corp. in recommending that the metal should be thoroughly soaked at 1550 to 1650° F. and then rapidly cooled. The temperature range of 1400 to 1600° F. is used by The Babcock & Wilcox Tube Co. for annealing cold worked tubing; the tubing is water quenched from the annealing temperature. Weldments have been stress relieved at a temperature of 1350° F., cooling by air blast or water quenching, depending on the shape and nature of the welded joint or structure.

Service Examinations and Data

In this section are summarized the conclusions of laboratory tests conducted by The Babcock & Wilcox Tube Co. and on pilot plant and plant exposure tests reported by the operating companies participating in the Metals Research Program.† These investigations studied the relative behavior and evaluate the general suitability of 27% chromium-iron under the actual conditions involved in the catalytic dehydrogenation process. Specifically, these conditions are:

1. Temperatures of from 900 to 1500° F.
2. Oxidizing conditions due to catalyst regeneration.
3. Reducing conditions due to the action of the catalyst whereby free hydrogen, carbon and unsaturated hydrocarbon compounds are formed.
4. The oxidizing condition and reducing condition alternately existing in the same reaction chamber or catalyst tube.

Table XII — Oxidation Rate (In. per 1000 Hr.) of Various Alloys After Plant Exposure in Butadiene Reactors*

Material	Center of Reactor Below Catalyst Bed				In Reactor Above Catalyst Bed
	No. 2 Reactor	No. 3 Reactor	No. 4 Reactor	No. 5 Reactor	No. 2 Reactor
18-8 Cr-Ni	0.0005	0.0001	0.0001	0.0002	0.0001
18-8 Cr-Ni-Cb	0.0004	0.0001	0.0001	0.0001	0.0002
16-13-3 Cr-Ni-Mo	0.0002	0.0001	Nil	0.0002	0.0001
18-13-3 Cr-Ni-Mo	0.0001	Nil	Nil	Nil	0.0001
18-8-2 Cr-Ni-Si	0.0001	Nil	0.0001	0.0001	0.0001
Inconel	0.0037	0.0010	0.0017	0.0025	—
25-12 Cr-Ni	Nil	Nil	Nil	Nil	0.0001
25-20 Cr-Ni	Nil	Nil	Nil	Nil	0.0001
27% Cr	Nil	Nil	Nil	Nil	0.0001
29-9 Cr-Ni	Nil	Nil	Nil	Nil	Nil
Copper	0.0016	0.0011	0.0012	0.0018	—
Aluminum Bronze	0.0027	0.0053	0.0050	0.0045	0.0059
12% Cr + 0.2% Al	0.0396	0.0225	0.0211	0.0298	—

*Data from plant exposure tests by Standard Oil Co. of California and Sun Oil Co.

5. The necessity that the reaction chamber or catalyst container be kept practically free of iron and iron oxide.

Pilot plant work has demonstrated that under the conditions employed in dehydrogenation of n-butane, some ferrous alloys suitable for high temperature service are attacked and generate a dust with undesirable catalytic effects. This dust

†For details see Section I in reference No. 133. The authors, titles and dates of these reports are listed on pages 1024 and 1028.

collects on the surface of the catalyst with the result that carbonaceous deposits are formed at an accelerated rate until finally the catalyst tube is plugged (see Fig. 25).

To study this problem, several companies — including Phillips Petroleum Co., Universal Oil Products Co., and Shell Oil Co. — carried out extensive laboratory and pilot plant tests using tubes of various chemical composition, such as 18-8 Cr-Ni, 23-1 Cr-Cu, 27 Cr-nitrogen, 25-20 Cr-Ni and 25-12 Cr-Ni. It was definitely established that the 27% chromium-nitrogen alloy best resisted dust formation and, in general, was well suited for the other conditions of catalytic dehydrogenation.

The superior resistance to corrosion in butadiene reactors of 27% chromium-iron, as compared with a great variety of Cr-Ni steels and nonferrous alloys, was demonstrated by plant exposure tests conducted by Standard Oil Co. of Calif. and Sun Oil Co. The data cited in Table XII clearly show that the oxidation rate was negligible and of the same order as that of the higher chromium-nickel steels.

Service examinations by The Babcock & Wilcox Tube Co. mainly involved 27% chromium-iron tubing removed from butane dehydrogenation service. An attempt was made to correlate the characteristics observed under actual service with the data established previously under carefully controlled laboratory conditions.

In general, it may be stated that embrittlement, due to sigma phase precipitation or "885° F. embrittlement" (or both), would appear to be the one most deleterious factor curtailing useful life in dehydrogenation plants. Sigma phase was discovered in straight chromium alloys[147] with as low a chromium content as 26% (see Fig. 13, 14 and 15, page 988) but was absent in some alloys[143] containing 28.5% chromium.

This finding confirms the fact that chemical composition alone is but *one* of the requisites for sigma formation, other and equally important ones being the operating temperature and the time of exposure. Residual stress is an accelerating factor, because it supplies energy to stimulate the metal's phase reaction.

The data in Table XIII illustrate this point, since they show conclusively that the "sigma factor" computed as the sum of the principal alloying elements (Cr+Mn+Si+Ni) does not correlate with the degree of sigma formation. On the other hand, the hardness data in the last column of this table show that a definite relationship exists between degree of sigma formation and hardness — or rather between sigma and temperature, inasmuch as hardness, in these samples, is a direct function of the temperature they have endured in service.

In this connection, one of the more apparent conclusions from these service examinations was that the deleterious effect of sigma would be adequately counteracted, and therefore the life of the tubes would be extended considerably, by periodic removal of the tubes from the dehydrogenation reactor furnace for reannealing before replacement (wherever it is feasible so to do). In some installations this can fortunately be done *in situ* before shutdown of the catalyst case, thus annealing the metal. Embrittlement at 885° F. is circumvented by quickly cooling the case through the temperature range from 1200 to about 700° F.

Apart from its effect on the formation of

Table XIII — Chemical Composition Versus Hardness and Amount of Sigma Constituent in 27% Chromium-Iron Tubes After Plant Service[143]

Sample No.	Average Composition						Sigma Formation Factor*	Vickers Hardness†
	C	Mn	Si	Cr	Ni	N_2		
				No sigma‡				
6	0.145	0.575	0.28	28.52	0.28	0.219	29.655	190
				Very light sigma‡				
1	0.09	0.51	0.39	26.18	0.19	0.146	27.270	184
14	0.135	0.615	0.52	26.96	0.155	0.154	28.250	to
18	0.14	0.455	0.575	27.93	0.56	0.19	29.520	215
				Light sigma‡				
5	0.12	0.655	0.425	28.81	0.415	0.227	30.305	211
13	0.135	0.45	0.475	26.99	0.41	0.17	28.325	to
9	0.14	0.99	0.435	26.85	0.455	0.161	28.730	228
				Medium sigma‡				
4	0.135	0.82	0.44	28.49	0.37	0.161	30.12	220 to
17	0.095	0.55	0.535	27.55	0.42	0.148	29.055	250
				Heavy sigma‡				
2	0.115	0.89	0.485	28.39	0.47	0.16	30.235	
3	0.135	0.87	0.41	27.76	0.785	0.183	29.825	
7	0.145	0.995	0.455	27.58	0.535	0.162	29.565	
8	0.17	0.845	0.41	26.63	0.34	0.182	28.225	
10	0.115	0.88	0.48	27.44	0.265	0.136	29.065	231
11	0.12	0.93	0.475	27.32	0.29	0.166	29.015	to
12	0.125	0.965	0.455	27.09	0.35	0.171	28.860	313
15	0.135	0.965	0.415	27.62	0.355	0.199	29.355	
16	0.16	0.42	0.565	27.89	0.565	0.197	29.440	
19	0.13	0.545	0.40	28.47	0.35	0.209	27.765	

*Sigma factor = sum of Cr+Mn+Si+Ni. †30-kg. load.

‡Amount of sigma constituent determined by microscopic examination of samples removed from dehydrogenation service.

sigma phase in 27% chromium-iron alloys, temperature is directly responsible also for other types of failures such as creep and stress-rupture. Incipient fusion of the metal as a result of contact between the catalyst, carbon and tube surface at very high temperatures (see Fig. 26, 27 and 28) also has occasioned failures in service. Such inadvertently high temperatures result from high oxygen in the regeneration gas; overheating also carburizes the metal with corresponding loss of chemical inertness in this process.

Summary and Conclusions

The foregoing sections of this compilation have supplied information, from various sources, on the properties, physical constants, metallurgical features (including embrittlement phenomena), and behavior of 27% chromium-iron alloy with respect to chemical inertness under conditions encountered in butane dehydrogenation. An understanding of the characteristics of this high chromium alloy, especially its grain growth on welding, lack of transformations during heat treatment, its extreme notch sensitivity, and its tendency to become embrittled within the range of temperatures encountered in the dehydrogenation process, warrant several suggestions in the interest of securing satisfactory service and of minimizing maintenance and breakage. Inherently the alloy offers good chemical resistance to the alternate reducing-oxidizing cycles encountered in the dehydrogenation of hydrocarbons between 900 and 1350° F. by the catalytic process.

Up to the present, its use in these processes has been confined primarily to preheater tubes, process piping or liners for such piping, headers, snake tubes, and catalyst tubing and fittings in dehydrogenation plants using unit or tubular type catalyst case constructions. Some of the alloy also has been used for protection tubes of thermocouples measuring catalyst bed temperatures, for catalyst support screens and plates in vessel reactors, and for sheets or baffles in catalyst vessels to prevent gas infiltration back of the refractory linings. The following suggestions, based on theoretical considerations and upon practical experience to date, may prove helpful in securing satisfactory service.

Welding Repairs — In welding, use of a nickel-bearing austenitic or partially austenitic welding electrode (such as 25-20 Cr-Ni or 27-4 Cr-Ni) will reduce heat affected zones adjacent to the welds and provide better ductility in the weld metal than can be obtained with electrodes of high chromium-nitrogen. Preheating between 400 and 800° F., especially on heavy wall tubes or thick material, is recommended to avoid weld cracking. Preheating has been dispensed with on thin gage metal as well as in certain very simple forms of butt-joints without encountering underbead cracking; to avoid undue difficulties, however, it is usually desirable to preheat before welding. All welds should be stress relieved at 1300° F. min. immediately after welding. Cooling after stress relieving should be as rapid as practicable. Backing-up rings should be used on most joints to avoid the possibility of poor fusion with resulting notches at the weld root.

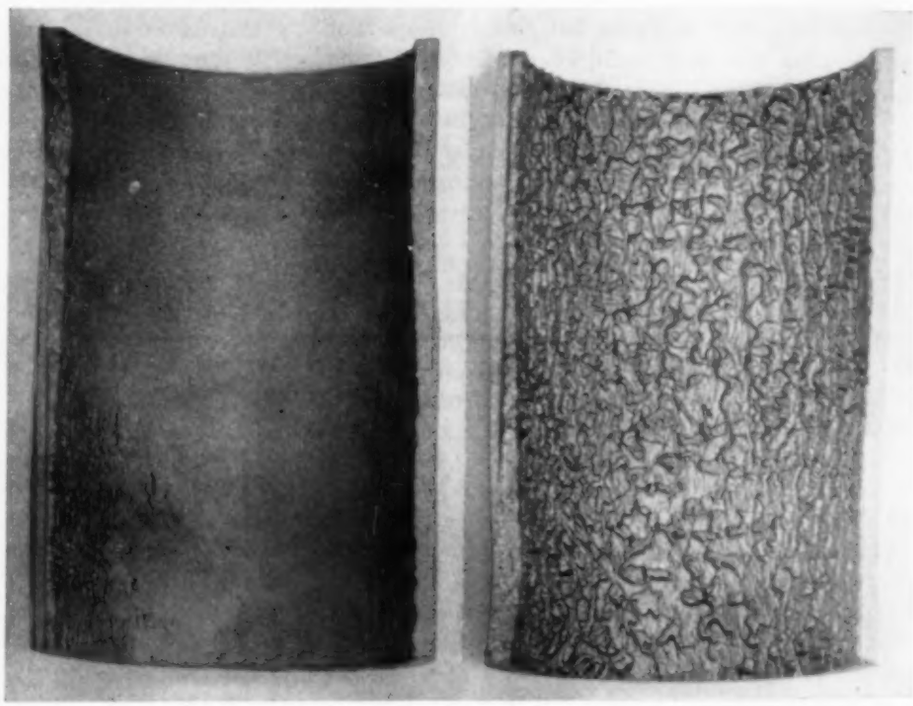


Fig. 26 — Bulged Sample of Fig. 25, Split and Cleaned by Sandblasting[142]

Avoidance of Notches — Avoid sharp notches and re-entrant angles in design or repair. In machining operations, allow for large radii to avoid stress concentration. In sawing or other machining or cutting operations, avoid under-

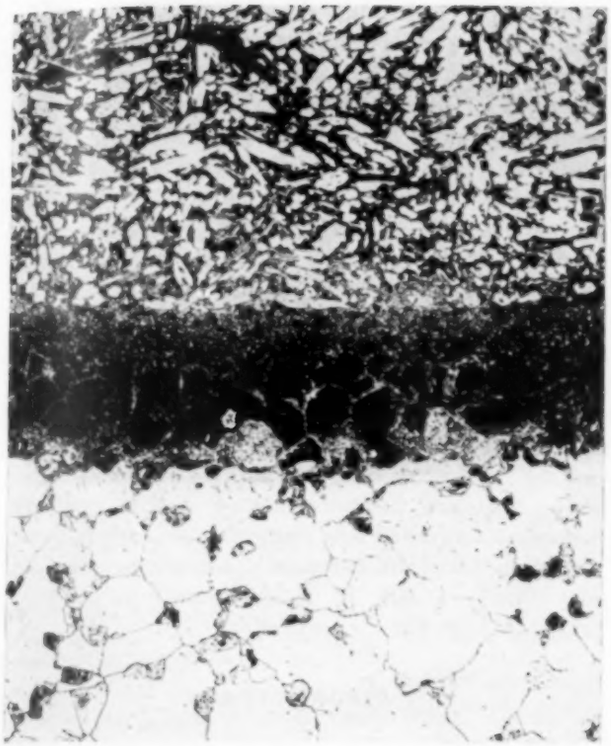


Fig. 27 — Microstructure Near Inner Surface of Roughened Tube of Fig. 26. Note cast or fused high carbon metal at top (inside), then narrow layer of metal carburized by diffusion, and base metal at bottom[142]. *100 ×*

cutting such as might give rise to notches. Avoid folds, nicks, scratches, stamping of numbers, or other mechanical abuse of surfaces.

Elimination of Shock Stresses — Shock stresses (pounding, hammering, vibration) should be avoided, particularly if the metal is at ambient temperature. Operations involving shock or impact should be performed, if required, when the metal is at 500° F. or higher, where some toughness and ductility will be restored. This is particularly necessary if the metal has been allowed to cool slowly through the embrittling temperatures.

Lubrication, Copper Gaskets — Use of copper gaskets on certain types of flanges and hand hole fitting seats has helped maintain tight, leak-proof joints. Graphite, applied to threads and contact areas of bolted fittings (Unibolt), has reduced galling and sticking. Without such lubrication, joints, fittings or tubes may fracture if shock is applied to loosen them.

Elimination of 885° F. Embrittlement — Cooling, after service, should be relatively rapid through the 1100 to 700° F. range so as to avoid the so-called 885° F. or 475° C. brittleness with its concomitant loss of ductility. Cooling may sometimes be done by circulating cold air, opening dampers or similar manipulation of the equipment. Below 700° F. (371° C.) the cooling should be more moderate to avoid undue mechanical (thermal) stresses in structures of complicated design.

Heat Treatment to Remove Sigma Phase — Sigma phase, a brittle intermetallic iron-chromium compound, forms in alloys containing over about 25% chromium on long heating in the range from about 950 to about 1250° F. Precipitation of this microstructural phase causes increased hardness and drastic loss of ductile properties. It cannot be removed by quickly cooling from 1100° F., as suffices for correction of the 885° F. embrittlement. Furthermore, after appreciable embrittlement by sigma phase, the alloy does not recover ductility at moderate temperature and it may easily be broken by shock or deformation when in such embrittled condition. Therefore, wherever practical, a heat treatment for removing sigma phase should be instituted to transform the precipitated sigma phase back to ductile ferrite before the structure is cooled down. This can be done by heating for not less than one hour at a minimum temperature of 1500° F. After putting the sigma phase back into solid solution at the higher temperature, the metal should be quickly cooled through the range 1100 to 700° F. so as to avoid the 885° F. embrittlement. Sigma phase does not

Fig. 28 — Brittle Fracture in 27% Cr-Fe Tube Removed From Dehydrogenation Service[142]

appear to detract from the chemical resistance of the alloy for the dehydrogenation process; its primary effect is to alter seriously the physical properties. The heat treatment recommended above may be done *in situ* if the equipment is designed to reach and withstand 1500° F., the minimum temperature indicated. Otherwise, the parts may be disassembled and treated in furnaces especially provided for the purpose. The main benefits derived are more ready handling and reduction of failures (cracking).

Gross Overheating — Metal dust and ordinary steel articles which are inadvertently left in equipment, may lead to excessive carbon formation and plugging of catalyst tubes. High conversion temperatures may stimulate carbon formation, and low regeneration gas pressure may result in incomplete reactivation leaving tubes in a plugged condition. Volume increase from the coke formed causes excessive stressing of the tube metal with bulging or fracture. Conversely, regeneration gas high in oxygen content may cause excessive temperature and may actually melt the tube wall and catalyst support grid. Likewise, leaks or cracks in equipment can lead to excessive temperature from fires caused thereby.

When temperatures exceed about 1700° F. the metal structure may be altered by carbon absorption (carburization) with loss of chemical resistance. This is due to chemical combination of chromium and carbon to form carbides, thus depleting the chromium content effective in solid solution with iron to resist oxidation. Excessive temperatures may also result in grain growth, intercrystalline fracture (and lowered melting point, if the metal has been carburized).

Regeneration procedures should be controlled at all times to prevent overheating. An excessive temperature would be considered one that is high enough for carburization of the metal; this will occur before any temperature necessary for substantial grain growth is attained in the nitrogen-bearing alloy. This probably means temperatures in excess of about 1700° F., although lower temperatures should be maintained if tubes or equipment show any tendency for bulging or sagging. Ordinarily, the maximum temperature reached during normal operations of dehydrogenation should not exceed about 1350° F., except during heat treatment for sigma phase removal.

The remarks in the preceding paragraphs relate mainly to the alloy as applied in the dehydrogenation process. Some of them are also applicable when the alloy is in other uses — particularly as regards welding, notch sensitivity and cooling to avoid 885° F. embrittlement. Sigma phase will not be encountered during ordinary corrosion service at low temperatures, nor in usual oxidation resistant services — as, for example, in soot blowers — for the latter operate at temperatures too high for its formation.

Acknowledgments

The author wishes to acknowledge the helpful assistance of his associates in the research department of The Babcock & Wilcox Tube Co. Special thanks are due M. A. Cordovi for assistance in preparation of the manuscript, to Z. E. Olzak for conducting many of the investigations, to the company for permission to include certain unpublished data, and to the several oil companies joining in the research program for much laboratory and plant service data on the performance of alloys in petroleum butadiene service. Special appreciation is due M. E. Holmberg of the engineering department, Phillips Petroleum Co.; K. V. King, materials engineer, Standard Oil Co. of Calif.; and A. B. Bagsar of the metallurgical department, Sun Oil Co.

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(Continued on page 1016)

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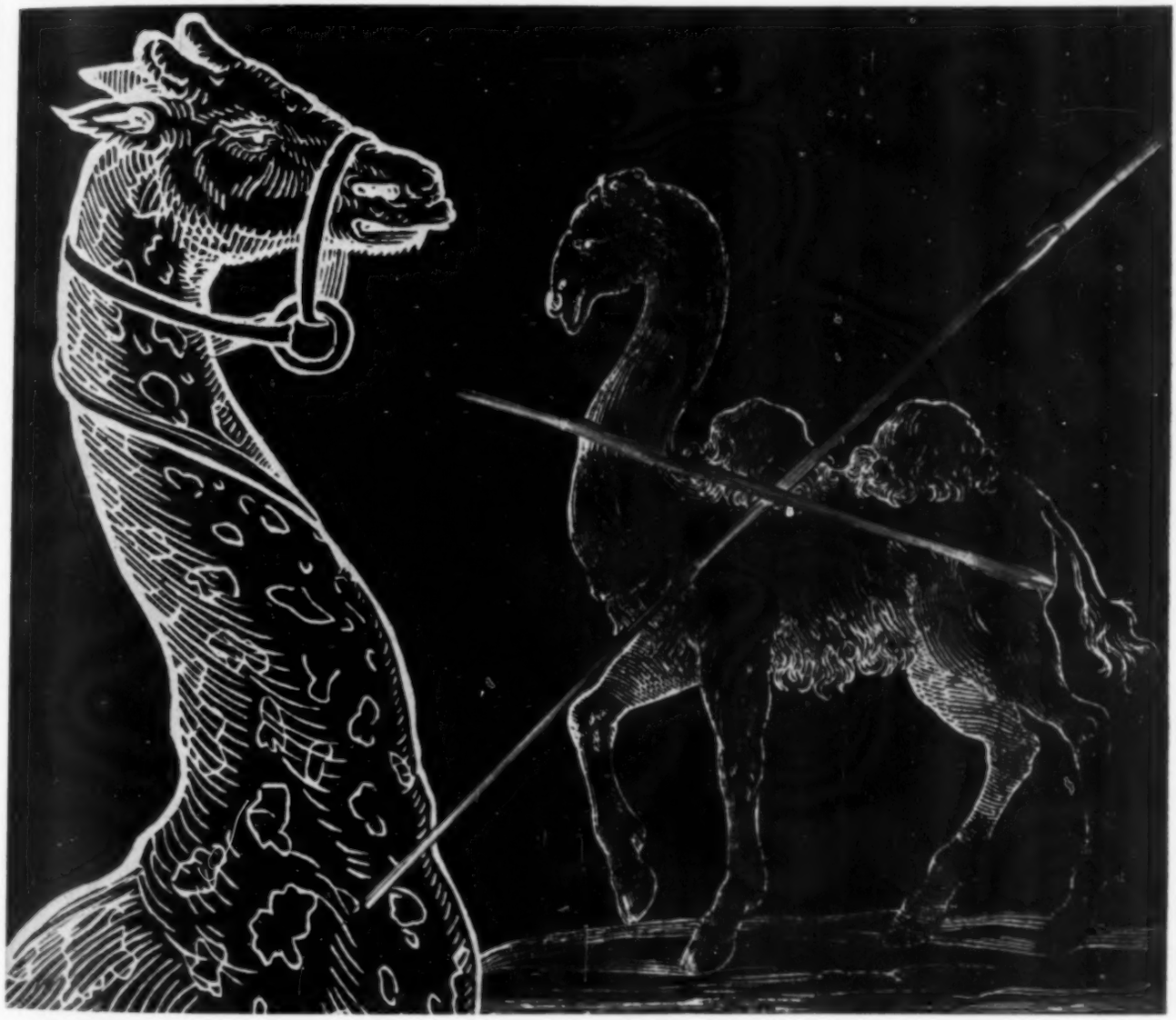

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Personals

WALTER R. GIESE, formerly of the Nash-Kelvinator Corp., is now employed as a metallurgical engineer for the E. I. du Pont de Nemours & Co., Inc., experimental station at Wilmington, Del.

After being placed on inactive duty by the U. S. Navy, ROBERT L. NICHOLS has returned to the metallurgical dept. of the steel and tube division of Timken Roller Bearing Co., Canton, Ohio.

CYRIL STANLEY SMITH was awarded a Medal of Merit citation for his role in the production of the atomic bomb. Now director of the Institute for the Study of Metals, University of Chicago, Mr. Smith was associate division leader at the Los Alamos Laboratory.

LEONARD F. WARTMAN, GEORGE R. WLODYGA, RICHARD KIRCHMAYER, and JOHN R. KARPOWITZ have founded the Austin Gray Iron Foundry in Sheboygan, Wis. Mr. Wartman is president of the foundry, Mr. Kirchmayer vice-president, Mr. Karpowitz secretary, and Mr. Wlodyga is treasurer and director of research.

C. B. WILLMORE will be chief metallurgist and will design and direct the new research laboratories of William F. Jobbins, Inc., Aurora, Ill. Mr. Willmore was formerly assistant chief of process metallurgy research at the New Kensington plant of the Aluminum Co. of America.

R. F. MATHER, formerly chief metallurgist with Willys-Overland Motors, Inc., is now chief metallurgist at the Kaiser-Frazer Corp., Willow Run, Mich.

PAUL D. GORSUCH is now employed as a metallurgical engineer with the Schenectady works laboratory of the General Electric Co.

LT. COMDR. HARRY J. HUESTER is Navy technical representative on the Army Air Force tropical science mission intended to study conditions in the Pacific tropics affecting deterioration of materiel.

R. GENDERS has recently relinquished the post of superintendent, Technical Applications of Metals, Ministry of Supply, England, to take up private practice as a consultant on materials and research. He will still act as metallurgical advisor to the Ministry of Supply.

EMIL GATHMANN, JR. and C. G. STEPHENS have organized the Gathmann Industral Corp. in Baltimore, Md., a firm of metallurgical and industrial consultants. HOLDEN R. HOUGHTON, formerly president of Houghton and Renoff, and ALBERT STARGARDTER, formerly chief metallurgist, Eastern Stainless Steel Corp., are associated with them.

The Ordnance Division has awarded a "Certificate of Commendation" to EDWIN K. SMITH "for outstanding performance of duty while serving as chief, materiel section, engineering and manufacturing division, Office of the Chief of Ordnance, Detroit."

MERVIN L. REYNOLDS has left P. R. Mallory and Co. to accept the position of technical representative with William Lynn Chemical Co. of Indianapolis.

GEORGE B. WATERHOUSE, past president, professor emeritus at Massachusetts Institute of Technology, was selected to deliver the first Hatfield Memorial Lecture at the annual general meeting of the British Iron and Steel Institute in London on May 1.

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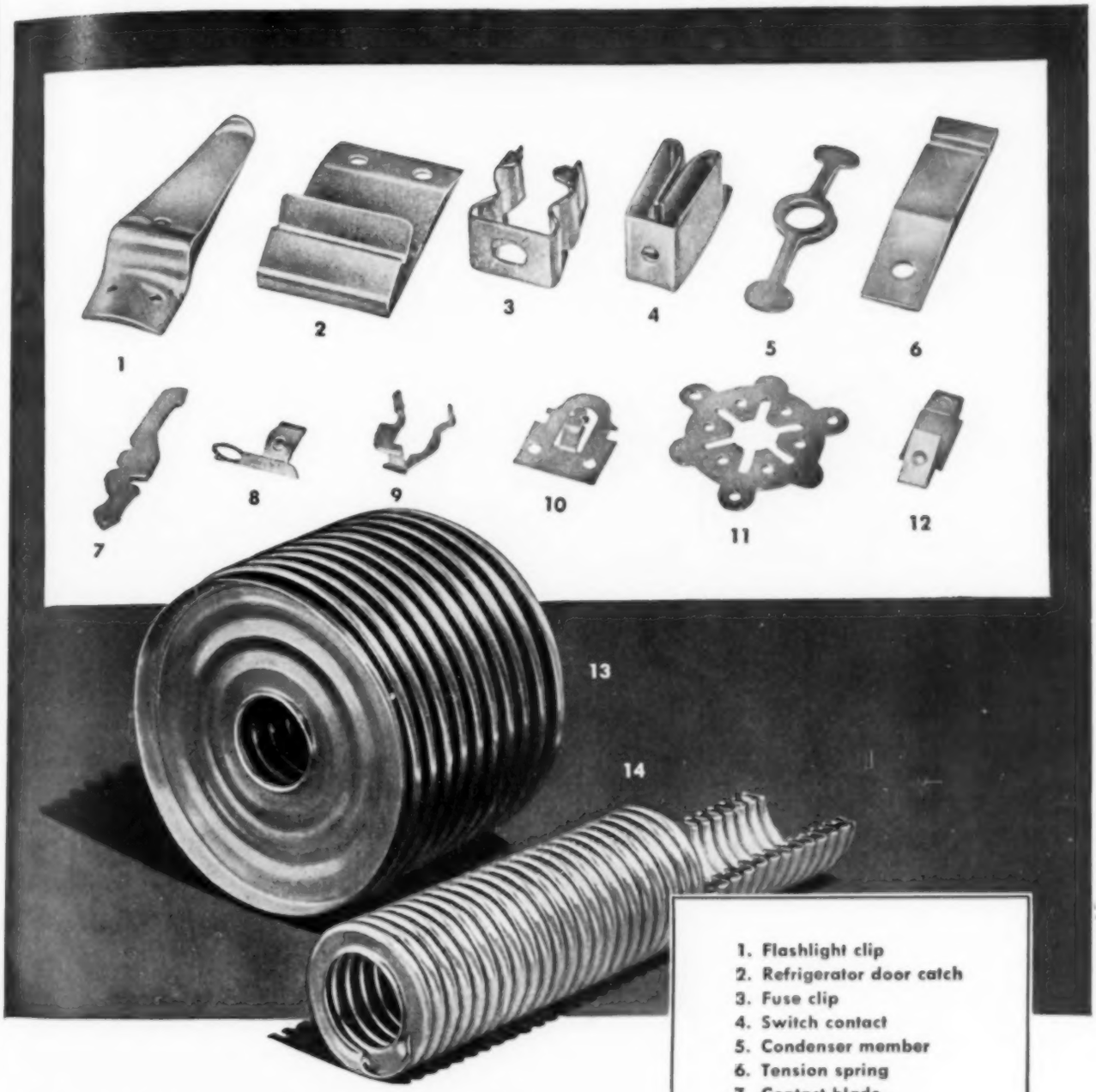

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Personals

JULIAN D. CAREY, JR., M, formerly with Wright Aeronautical Corp., is now a metallurgist for General Electric Co. in the Bridgeport, Conn., works laboratory.

GUILLIAM H. CLAMER M, president of Ajax Metal Co. and associated companies, is to deliver the American Foundrymen's Association Foundation Lecture at the convention May 6 to 10.

Appointment of the following men to positions in the newly organized ingot division, Louisville, Ky., of Reynolds Metals Co. has been announced: R. G. ROSHONG M, formerly technical advisor of the aluminum division, is product manager; T. D. STAY M, formerly of National Transit Pump & Machine Co., Aluminum Castings Co. and the Aluminum Co. of America, is technical advisor; P. C. BECK M, who before joining Reynolds Metals was with the Aluminum Co. of America, is a field service engineer.

EDWARD P. GEARY M has formed the Geary Stainless Steel Co., sales service engineers, in Baltimore, Md. Mr. Geary was previously assistant vice-president in charge of sales of Rustless Iron and Steel Corp. The Chicago office of Geary Sales will be under the supervision of ROBERT L. SPRINGER M, who was also formerly associated with Rustless.

H. A. VAN HALA M has been transferred by the Bristol Co. from the Birmingham branch office to the Cleveland branch office.

The American Foundrymen's Association announces the following awards to be made at the May convention of the association: HYMAN BORNSTEIN M, director of the testing and research laboratories of Deere & Co., Moline, Ill., is to receive the William H. McFadden Gold Medal for his many "outstanding contributions to the foundry industry, particularly in the field of gray cast iron"; HOWARD F. TAYLOR M, formerly with Naval Research Laboratory, Washington, D. C., and now research associate at Massachusetts Institute of Technology, Cambridge, Mass., will receive the first Peter L. Simpson Memorial Gold Medal; HAROLD J. ROAST M, vice-president of Canadian Bronze Co., Ltd., Montreal, will receive an honorary membership for his contributions to castings industry literature and cooperative enterprise.

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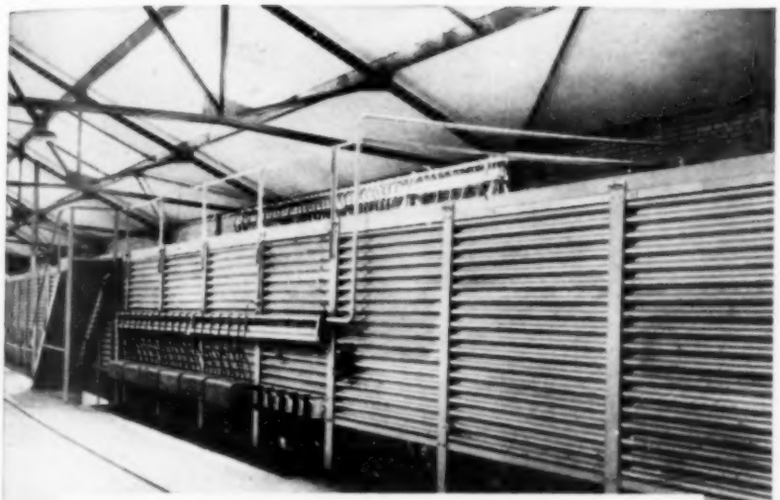

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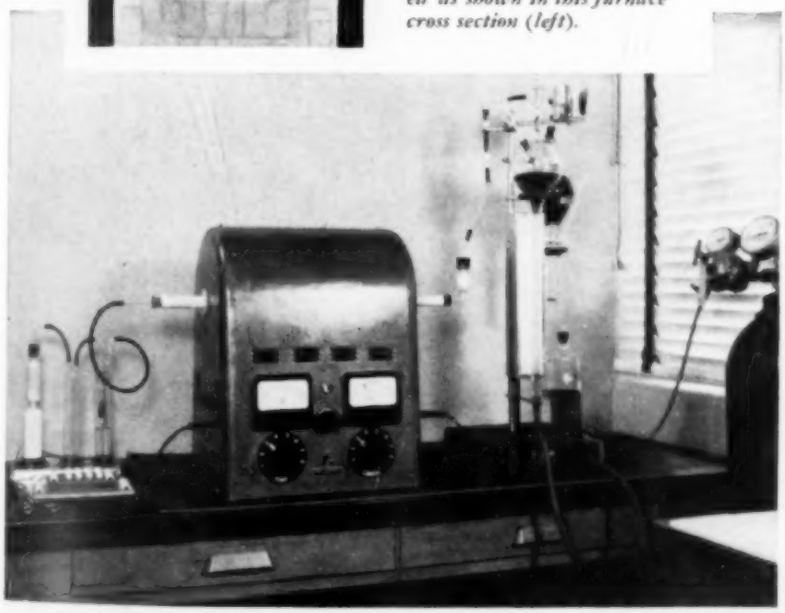

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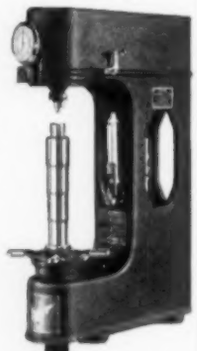

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Personals

PAUL A. SHOUDY, formerly with the Kirsten Pipe Co., has joined the staff of the Aero-Kool Pipe Co., Seattle, Wash., as research and production engineer.

ELI MULLIN has established his own office practicing patent, trademark and copyright law in Chicago. He was formerly associated with Carnegie-Illinois Steel Corp. as metallurgical engineer, and with the patent law firm of Wallace and Cannon.

NORMAN A. MATTHEWS, formerly at the Watertown Arsenal, has been appointed works metallurgist of Electro-Alloys Division of the American Brake Shoe Co., New York City.

A. R. DEIR is now assistant metallurgist at Noranda Mines, Ltd., Noranda, Quebec, Canada. He was previously an engineering officer in the Royal Canadian Air Force.

HOWARD L. FRANKS, formerly with Carpenter Steel Co., has been appointed sales manager of the specialties division of Charles Fischer Spring Co., Brooklyn, N. Y.

DON A. LAWLESS has been appointed to direct the new technical service department of the aluminum sales division of Reynolds Metals Co. Mr. Lawless has been material engineer in charge of the specifications section, metallurgical department of Reynolds Metals.

Bliss & Laughlin, Inc., Harvey, Ill., announces that HARRY M. CLARKE has been appointed assistant to the president. He has been associated with Bliss & Laughlin in a sales and metallurgical engineering capacity.

GERALD L. BRITTON is to be in charge of the new sales and service office of the Brown Instrument Co., division of Minneapolis-Honeywell Regulator Co., Rochester, N. Y.

After being released by the Army Engineer Corps, ARNE HEDSTROM has returned to Vanadium-Alloys Steel Co. and will be head of the new Indianapolis, Ind., sales office.

Bryant Heater Co., Cleveland, announces that LAWRENCE R. FOOTE has been appointed industrial application specialist for the eastern sales area with headquarters in New York City.

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In addition, Kodak supplies these three types of industrial x-ray film:

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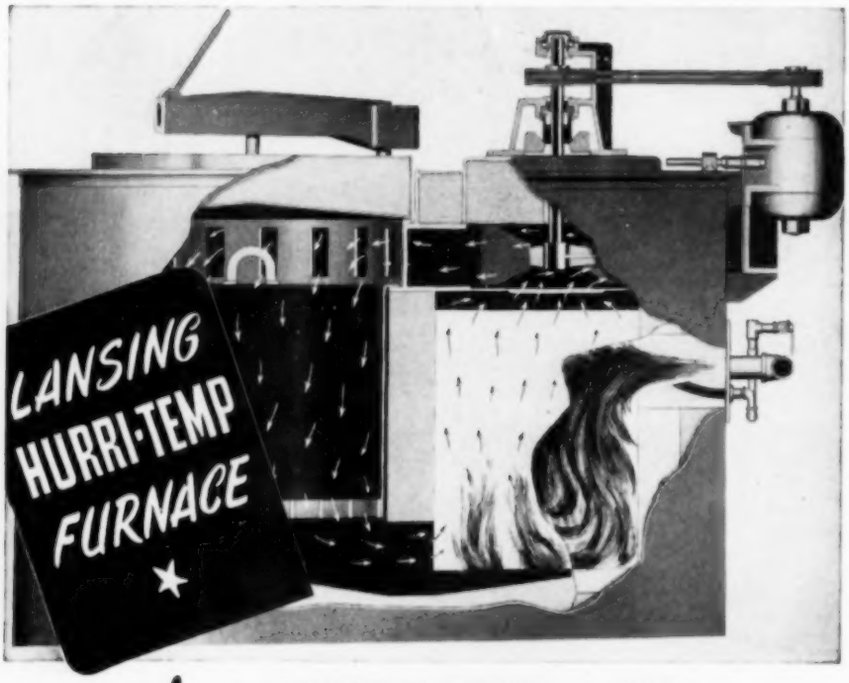

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Thorough, rapid heat penetration of densest loads. Gases from Lansing burners are drawn from specially lined combustion chamber and circulated through work by high speed fan.

Rugged blower unit, mounted in vertical position, has oversize radial and thrust ball bearings. Blower system is self contained, accessible, and easily removed as a complete unit. Motor, installed away from heat, is protected and quickly serviced.

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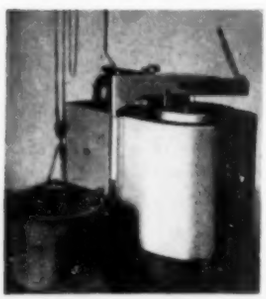

LANSING ENGINEERING COMPANY
934-36 Clark Street - LANSING 6, MICHIGAN

Personals

M. L. MEYERS, formerly with Studebaker Corp. and General Electric Co., is now vice-president of the new firm, Kokomo Foundry Corp., Kokomo, Ind.

KENNETH A. MOODY has been appointed training director for all American Steel & Wire Co. plants following his release from military service.

F. R. HENSEL, who has been chief metallurgical engineer of P. R. Mallory & Co., Inc., Indianapolis, for 12 years, has been elected vice-president in charge of engineering.

After three years' service with the Army Engineering Corps, HANS J. HEINE is now metallurgical engineer and metallographer with the Rockwell Mfg. Co., East Liberty, Pa., serving the Merco Nordstrom Valve Division, the Pittsburgh Equitable Meter Division and the Monessen Foundry Division.

The Lithium Co. announces the appointment of CARL O. ANDERSON as district sales manager for northern Ohio, western New York and western Pennsylvania.

LELAND E. HOUSEHOLDER has been appointed chief metallurgist of the Grand Rapids, Mich., extrusion plant recently acquired by the Reynolds Metals Co. Mr. Householder was with the Louisville, Ky., plant of Reynolds Metals previous to this appointment.

ELTON E. STAPLES has been made director of the new Cleveland district office of Hevi Duty Electric Co. Previous to his service with the U. S. Army, he was in charge of the Hevi Duty Chicago office.

A. Milne & Co. announces that ANDREWS W. CLARK will now be manager of the Hartford, Conn., office.

The Union Carbide and Carbon Corp. announces that RUSSELL FRANKS, for the past year chief metallurgist of the Union Carbide and Carbon Research Laboratories, Inc., has been appointed to the technical service and development department of Electro Metallurgical Co. in the Pittsburgh office.

ROBERT W. DOUGHERTY, recently discharged from the United States Navy, has been appointed assistant sales manager of the Lindberg Engineering Co., Chicago. He represented Lindberg in Detroit before entering the Navy.

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Heat Resistant and Stainless Steel Castings

Engineered to the Job . .

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BIRMINGHAM	**CLEVELAND**	**MILWAUKEE**	**PHILADELPHIA**	**ST. LOUIS**
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Stocks in Principal Metal-Working Centers

27% Cr-Fe

(Continued from page 1006)

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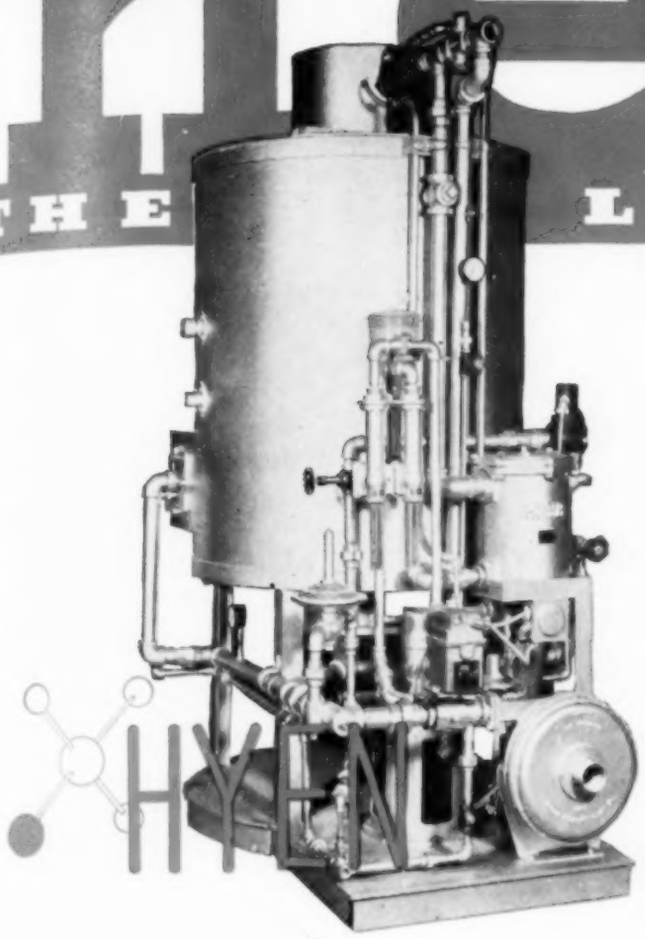
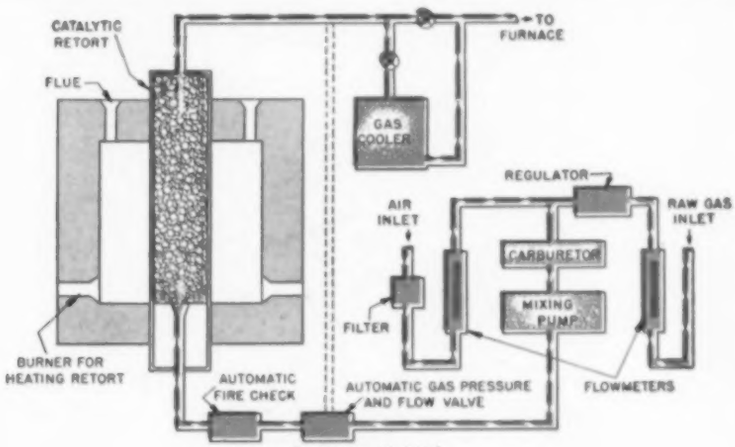
new

THE LINDBERG





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SCHEMATIC FLOW DIAGRAM FOR HYEN HYDRYZING GENERATOR

ATMOSPHERE GENERATOR

Fully Automatic! Endothermic!

***For* HARDENING - BRAZING - SINTERING *all medium and high carbon steels* WITHOUT DECARBURIZATION!**

Heat treaters have long wanted an atmosphere generator for production heat treating and brazing which was fully automatic, required no charcoal, did not have to be burned out periodically to remove soot, and could be depended on to produce a precision gas of consistent analysis month in and month out. Now after three and a half years of rigorous research—both in the laboratory and on 24-hour-a-day service on production lines, heat treaters agree that the Lindberg Hyen Generator meets all their requirements, and more, by enabling them to do a precise job easier, more simply, and with consistent results.

FULLY AUTOMATIC

Light the pilot, and from then on every phase of the generator is automatically controlled through push-buttons. Temperature, mixing, pressure and flow control are fully automatic. Gas ratios do not have to be readjusted when furnaces are cut in or out of the line.

NO SOOTING

Time is saved and production increased because high temperature cracking eliminates sooting. Thus, periodic cleaning and burn out periods are not required.

REFRACTORY RETORT

Higher cracking temperature than ever before possible because a new type refractory retort is used. Result is cleaner and drier atmosphere—No CO_2—Minus 10°F dewpoint—methane 1.0%.

FOUR SIZES

The Hyen Generator is made in 4 Standard sizes to fit any application—500, 750, 1500, and 2500 CFH. It can be used with your existing atmosphere furnaces. Because it is entirely automatic it can be located remote from furnaces.

GAS OPERATED

The Hyen Generator operates on city gas, natural, propane, or butane. It is an endothermic catalytic cracking type—uses only gas for both atmosphere and heating.

The HYEN derives its name from ENdothermic, and is one of the Lindberg line of four controlled atmosphere generators. The others are HYEX (EXothermic cracking), HYAM (AMmonia dissociation), and HYCO (high carbon monoxide).

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Write for the new 12 page Bulletin No. 190. "Lindberg Controlled Atmospheres"

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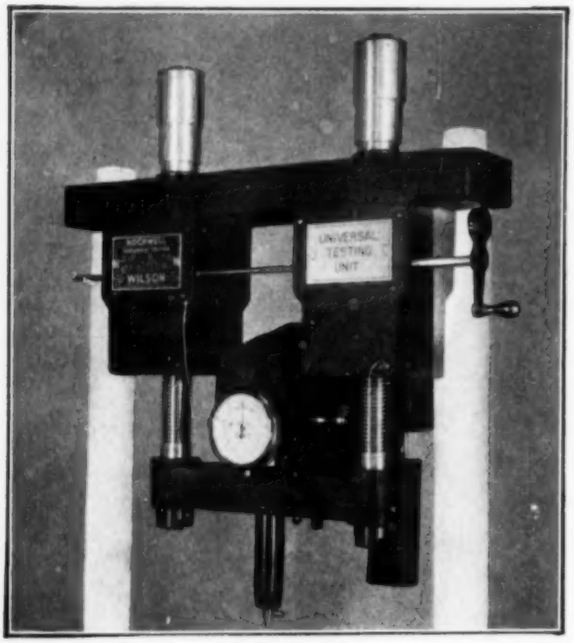

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MECHANICAL INSTRUMENT CO., INC.
367 Concord Avenue New York 54

27% Cr-Fe

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(Continued on page 1020)

No Flaking - No Chipping - No Peeling

when you use Anti-Corrosive IRIDITE as a Paint Base

Tests by several leading companies show that IRIDITE provides a firm base for paint and clear lacquer . . . as well as a corrosion-licking, protective finish . . . on zinc die cast, zinc plated, galvanized and cadmium plated products. IRIDITE keeps the zinc from fouling the paint . . . prevents formation of the soapy, chalky underlying film that destroys adhesion. IRIDITED products take paint readily . . . hold it permanently . . . without flaking, chipping or peeling!

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Moreover, IRIDITE is widely used as a final protective finish . . . on zinc, cadmium, galvanizing. A quick IRIDITE dip balks corrosion while adding eye-appeal to your products . . . since it is available in black, blue, green, red, bronze, olive drab and transparent IRIDITE bright. And to cut costs, use IRIDITE in combination with zinc plating to replace more expensive materials.

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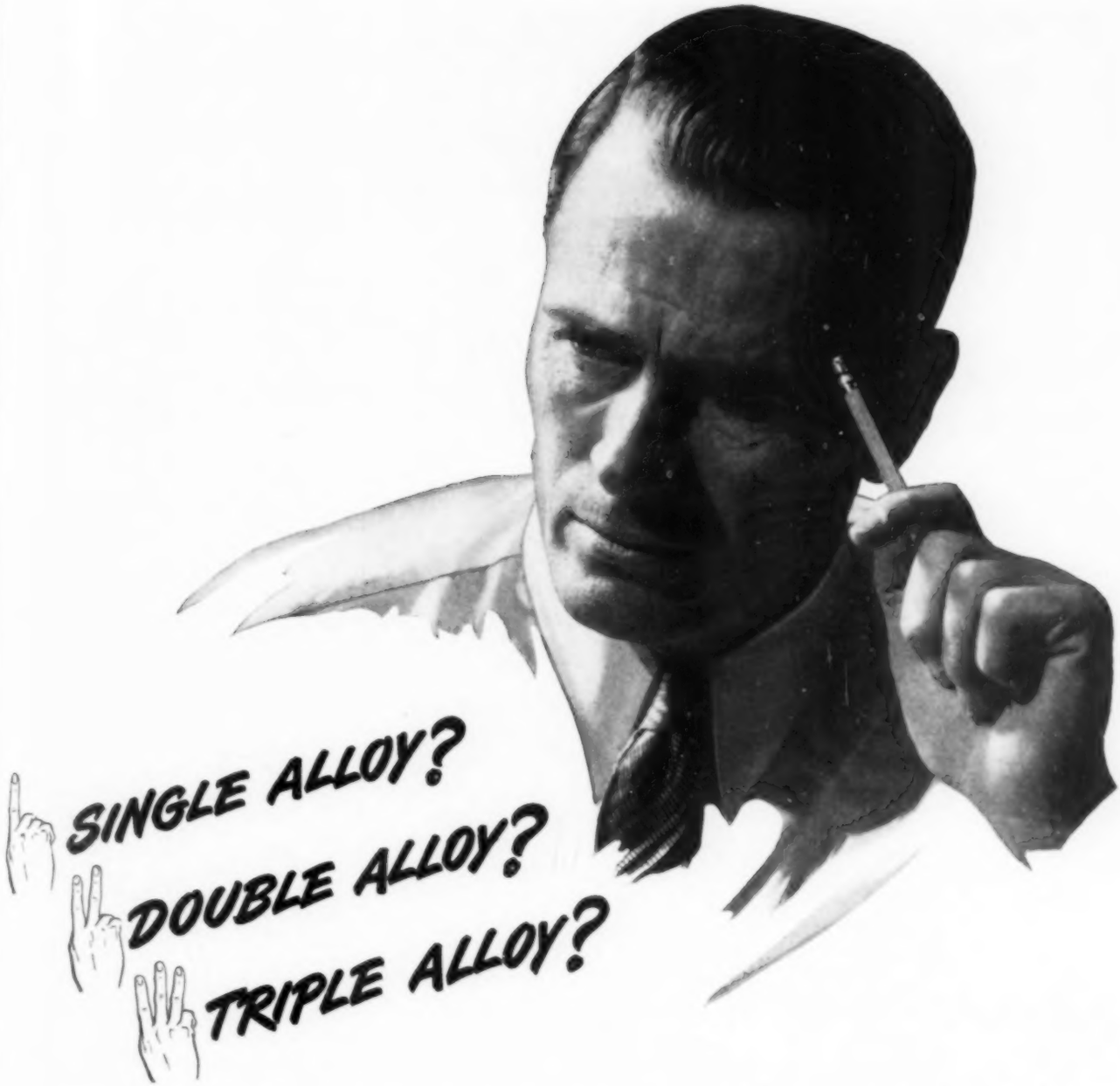

Are you looking for maximum toughness, the ultimate in hardenability per dollar of cost, or an analysis which lends itself to close hardenability control? These are but three of the many factors that govern your choice of alloy steels.

There is no one analysis that is best for all applications. But, whatever your problem, you can be sure of getting the *right* alloy steel from Bethlehem. Single, double, or triple . . . Bethlehem makes them all.

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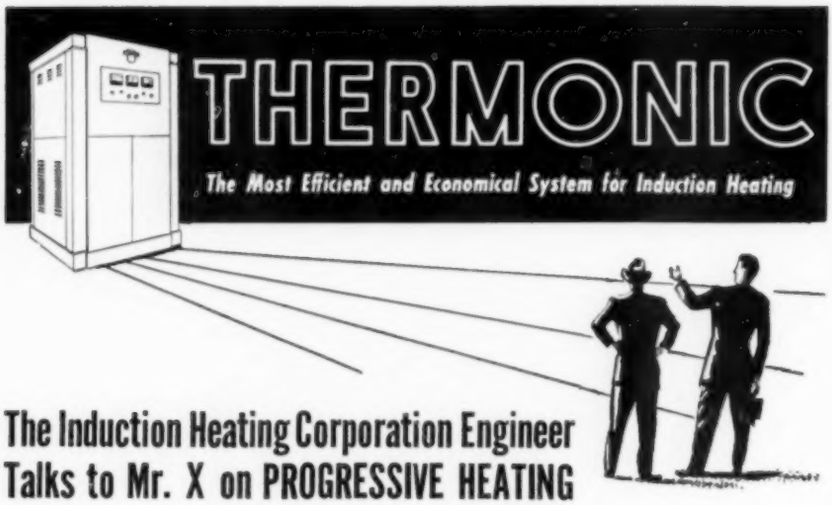

The Induction Heating Corporation Engineer Talks to Mr. X on PROGRESSIVE HEATING

MR. X . . . . Your recommendation of the electronic type of equipment for my job sounds pretty convincing, Mr. Engineer. There is something, however, I'd like you to clarify for me. You said that your THER-MONIC Induction Heating unit is suitable for hardening or annealing parts of comparatively small diameter. Does this mean that it can handle only small parts?

ENGINEER . . . . Not at all, Mr. X. Many large parts such as long shafts, bars, cylinders, and spindles are economically and efficiently heat-treated by our THER-MONIC units. You see, the heating of particularly large parts, requiring high power concentrations, is done progressively. This progressive heating is performed on only a small portion of the part at a time.

MR. X . . . . Just what do you mean by progressive heating?

ENGINEER . . . . I'll try to be more explicit. In progressive heating, large parts are fed gradually by a variable-feed mechanism, through a heating coil, having approximately the same shape as the part's cross-section. As the part moves automatically through the coil, a narrow band of its surface is heated to the proper temperature and immediately quenched by a spray of oil or water as it emerges from the coil. This process continues until the entire length of the part has been hardened.

MR. X . . . . But doesn't the part become warped and scale-formation take place as in flame-hardening?

ENGINEER . . . . No, sir. The extreme speed of this operation and the concentration of heat in only a small surface portion of the part at any one time prevent distortion. Then again, scale has not had time to form; and since only the temperature necessary for hardening is obtained, there is an absence of decarburization.

MR. X . . . . I see what you're driving at. But how can you control the degree of hardness obtained in a bar, for instance?

ENGINEER . . . . In continuous operations of this kind for a given generator, two factors regulate the depth of hardness of the bar being progressively hardened: namely, the rate of travel of the bar through the heating coil and the power input to the work. Obviously, the faster the bar travels at a given power input through the coil, the less the conduction of heat into the bar from the surface and hence, the less the depth of hardness.

MR. X . . . . I should imagine that heating would have to be very rapid to obtain a high rate of production on large parts?

ENGINEER . . . . That's right. Speed is an inherent characteristic of THER-MONIC Induction Heating. This is made possible by the ability of these units to transfer energy into metal at extremely high rates per unit of surface area. This is one of the limitations of ordinary furnace or torch heating. The full output of a THER-MONIC Generator can be concentrated into a single square inch of metal surface. These kilowatts of power, expressed in terms of B.T.U.'s per minute, give heat transfer rates which were previously unheard of in the heat-treating field.

MR. X . . . . Can you give me an idea of how induction heating compares with other methods of heating in regard to power concentration on a part?

ENGINEER . . . . Certainly, Mr. X. Rates of energy transfer from furnaces run in the order of 50 B.T.U.'s per minute per square inch of surface to be heated, while a torch flame can transfer energy at rates of slightly over 150 B.T.U.'s per minute per square inch. Both of these transfer rates are impeded by the formation of surface scale which prevents conduction of heat into the interior of a part. With induction heating, we speak of 10 to 25 kilowatts per square inch, which, translated into thermal terminology, are 500 to 1400 B.T.U.'s per minute per square inch!

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27% Cr-Fe

(Continued from page 1020)

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(Concluded on page 1028)

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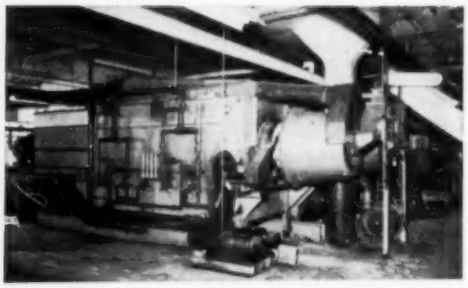
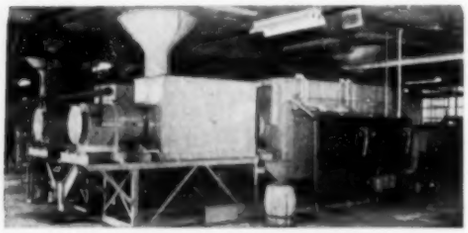
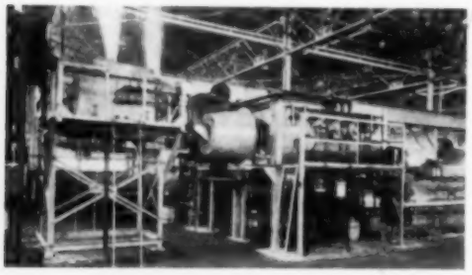


Gas-fired revolving retort furnace for annealing stampings; showing automatic feed to rotary charging hopper, and the long cooling zone.



Electric revolving retort furnace for annealing small pressed steel parts in a protective atmosphere; showing (in foreground) cooling zone with water sprays, and chute for feeding parts to floor below.



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27% Cr-Fe

(Continued from page 1024)

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Corrosion of Light Alloys

THE INFLUENCE of quenching temperature on the corrosion resistance of an aluminum-zinc-magnesium forging alloy with 4.5% Zn and 3.5% Mg provides the basis for a paper by W. Bungardt.* It has been observed by a number of German workers that stress corrosion of aluminum-rich Al-Zn-Mg forging alloys containing more than 5% combined zinc and magnesium depends not only on the cooling conditions after homogenization but also on the quenching temperature. The object of the present study was to determine to what degree the stress corrosion resistance of these alloys in the cold worked and quench-hardened *(Cont. on p. 1030)*

**Korrosion und Metallschutz*, V. 20, No. 3, March 1944, p. 119 to 124.

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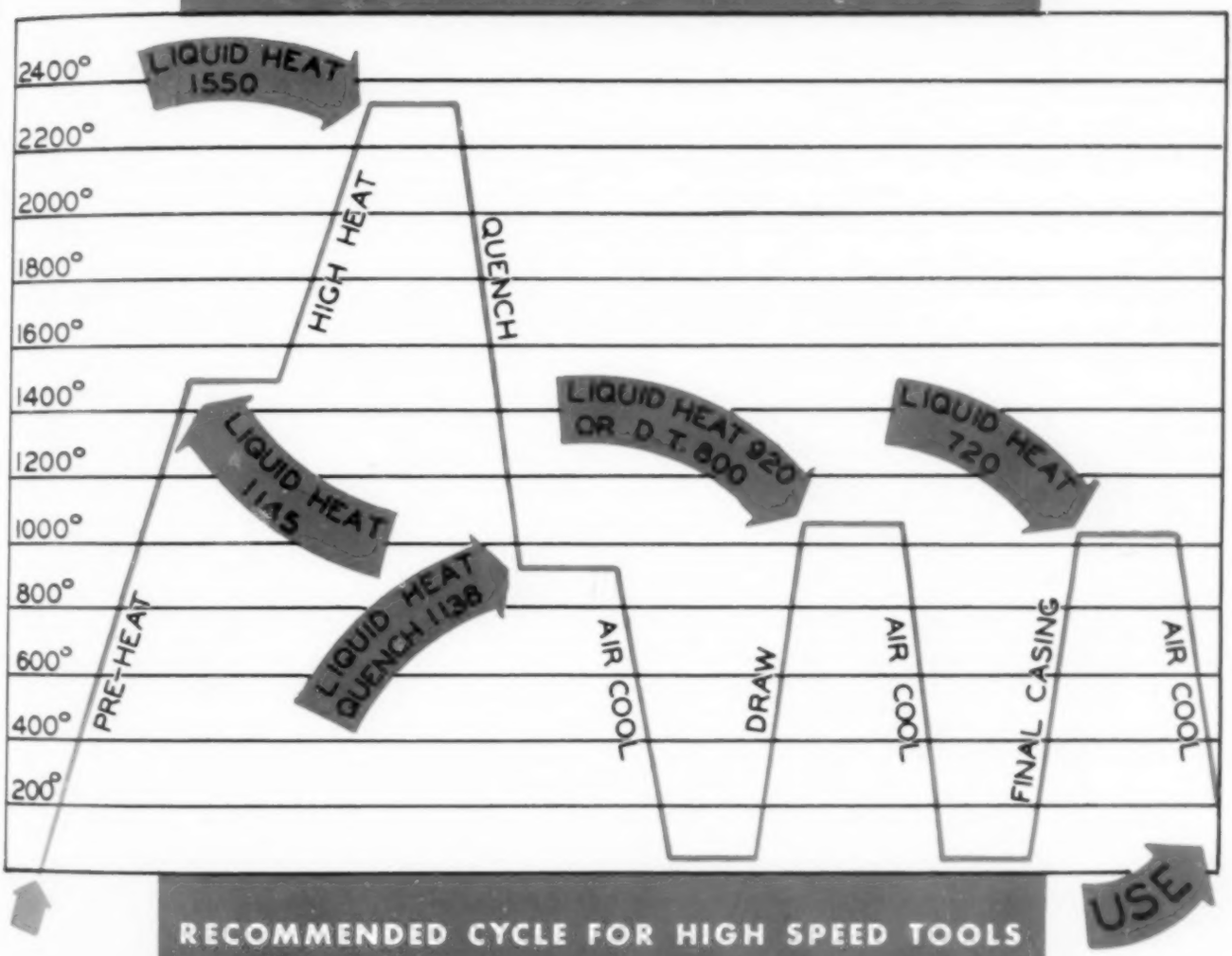

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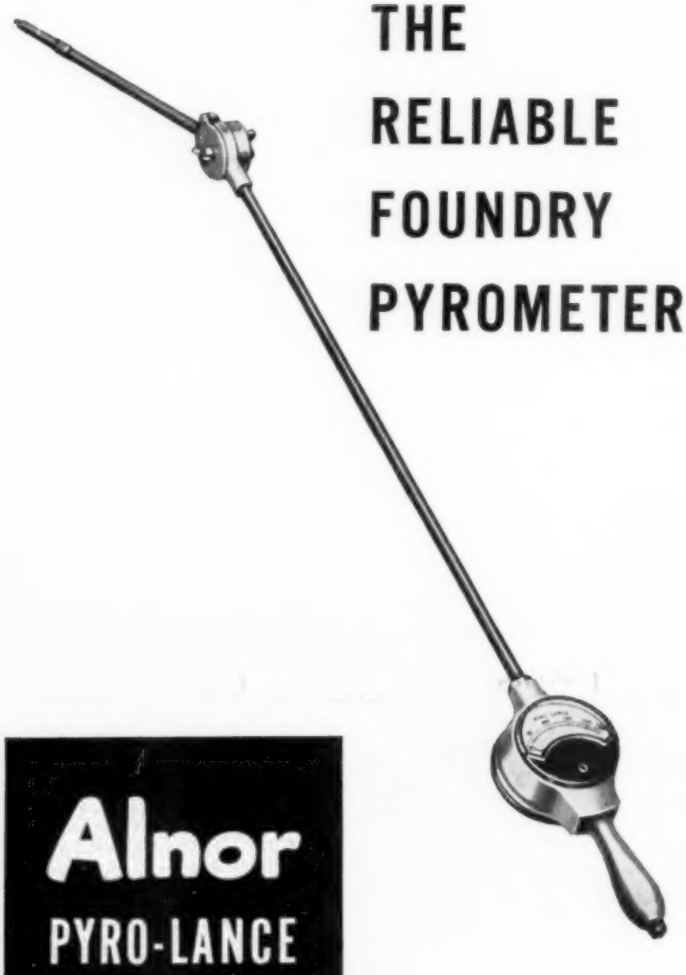

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Corrosion of Light Alloys

(From p. 1028) conditions could be improved by reducing the quenching temperature to just below the solution temperature.

Weathering tests involving intermittent immersion in synthetic sea water were made, and it was found that under certain conditions involving both room and high temperature aging, stress corrosion resistance was improved—naturally at the expense of the mechanical strength of the alloy. Comparative tests with air-cooled samples, nevertheless, clearly showed a superiority to this simplified heat treatment. The experimental results corresponded with changes in microstructure induced by the heat treatment.

According to previous work on alloys of this composition performed by Bungardt and Schaitberger, an increased susceptibility to stress corrosion is to be expected with water-quenched samples, subsequently age hardened at room temperature, after stress-relieving between 175 and 265° F. This was confirmed by the present authors, who found structures that were unstable even after prolonged age hardening at elevated temperatures and that tended to develop stress-corrosion cracks. It was found that a normalizing treatment at 212° F. after water quenching from 715° F. (even with short normalizing times) led to an appreciably higher stability against stress-corrosion phenomena, than when water quenching from 750° F. or even from temperatures above the solution point. On the other hand, it was found that with longer aging times, manipulating the quenching temperature and the annealing time had no effect.

Structural deterioration of aluminum and magnesium by corrosion, discussed in a recent paper by W. Patterson,★ is characterized not so much by surface attack and loss of metal as by penetration into the structure of the metal, without appreciable loss of metal. In this category belong stress-corrosion, intercrystalline and transcrystalline corrosion, pitting corrosion and, possibly, flaking.

The many observations that have been made indicate fairly clearly that deterioration phenomena of this nature depend, on the one hand, upon the nature of the phases present in the structure — the potential difference *(Cont. on page 1032)*

★*Korrosion und Metallschutz*, V. 20, No. 3, March 1944, p. 125 to 129.

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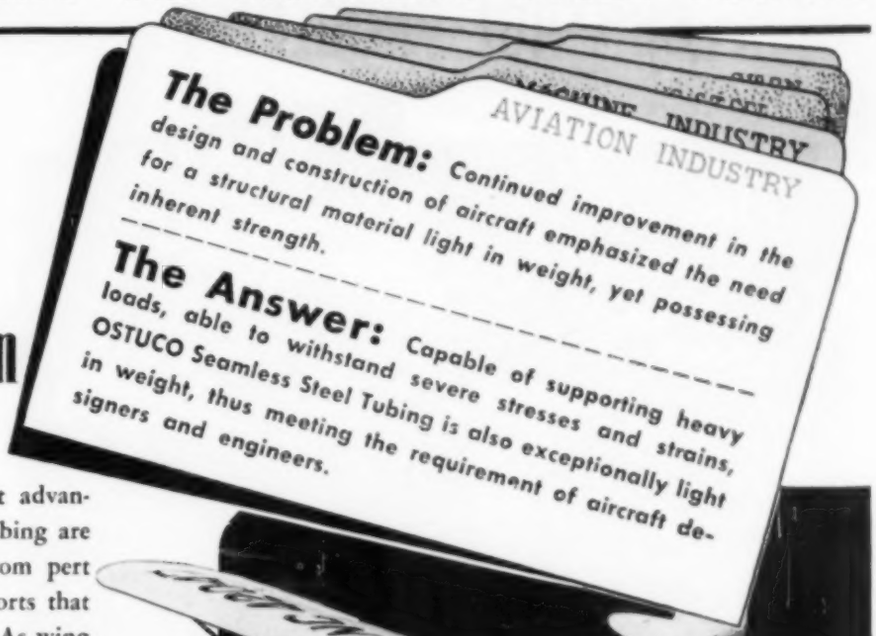
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Corrosion of Light Alloys

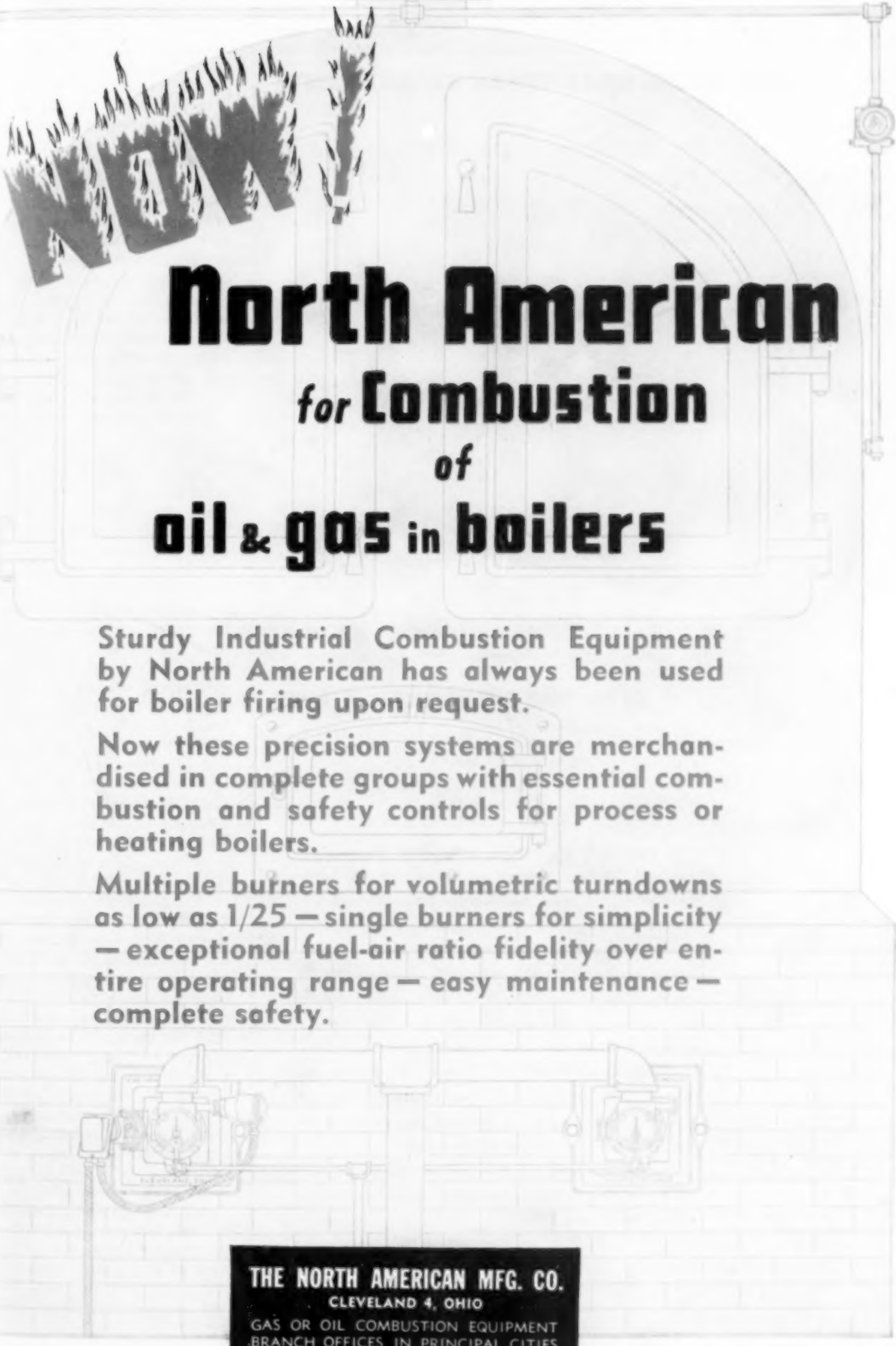
(From p. 1030) (electro-chemical) between the phases — and, on the other hand, on the arrangement of the phases in the structure. If the secondary constituents are less noble than the base metal, then on penetration of a corrosive agent, the less noble constituent will frequently be dissolved out. The surface is thus homogenized, so that further penetration of corrosion, to dissolve out further inclusions, is governed by the corrosion resistance of the solid solution. Conversely, if the secondary phase is more noble than the base metal, the latter will be dissolved out at the boundary phase limits, leading to a marked graining and pitting effect.

Numerous observations have confirmed the assumption that an essential condition for corrosive deterioration in light alloys is the presence of at least a two-phase structure with a definite phase arrangement. This phase arrangement may be of a varying nature, but the second phase should be dispersed through the solid solution in a network formation. Thus, resistance to stress corrosion may be effected by control of the precipitation from the super-saturated solid solution. Under controlled conditions of forming, annealing and alloying, material free from stress corrosion tendencies can be produced; likewise, material which will deteriorate under conditions of stress corrosion can also be made experimentally.

Many metallurgists assert that microscopic examination of the structure provides no criterion for judging the corrosion resistance of light alloys. The author's experience, however, is that examination under high magnification of certain Al-Mg alloys will definitely indicate instability with a certain structure or an unlimited service life with another type of structure.

Addition of certain alloying elements to aluminum alloys that are sensitive to stress corrosion suppresses the tendency to separation of the connected layers at the grain boundaries, supposedly by preventing diffusion. Zinc and manganese additions have this effect as well as the common stabilizers, chromium and vanadium. Corrosion proceeding parallel to these layers gives the peeling effect so often encountered in these alloys. A parallel example is provided by the inter-

(Continued on page 1034)



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Corrosion of Light Alloys

(Continued from page 1032)

crystalline stress corrosion of austenitic steels. Intercrystalline fissures appear in the microscopically homogeneous structure only in an area in which the gamma solid solution shows a marked tendency to break down into an alpha-plus-gamma phase which is stable at room temperature. In forming 18-8 steels, the martensite formed during the fabricating process leads to a peeling effect along the slip planes, similar to that mentioned with the Al-Zn-Mg alloys. All of these observations point to a two-phase structure beyond the resolving power of the microscope.

Likewise, the intercrystalline corrosion occasionally observed in magnesium alloys is obviously connected with such a two-phase structure with a definite phase arrangement. In forming magnesium alloys, precipitates appear on the slip planes, which subsequently provide corrosion paths through the crystallites. If, by a suitable heat treatment, these layers are homogenized on the slip planes, the tendency to stress corrosion then disappears.

Weld cracking in magnesium-manganese alloys with cerium additions is studied in a paper by H. Maeder and F. Laves.★ The strength properties of the Mg-Mn alloys are considerably improved by cerium additions, the cerium acting specifically to raise the elongation and strength at elevated temperatures. This is, however, accompanied by a deterioration in welding properties. Alloys with 0.2 to 0.3% Ce tend to pronounced crack formation on welding. Investigations have shown that the addition of about 1% aluminum to these alloys will entirely suppress the susceptibility to weld cracking.

Microscopic examination showed that the cerium was present as the compound $CeMg_9$, principally in the form of round grains. In the superheated, coarse-grained zone adjoining the weld seam, however, it occurs as a skin between the magnesium-rich solid solution. When aluminum is added to the alloy, a new type of crystal is formed, $CeAl_2$, finer grained than the $CeMg_9$. In the cerium alloy with the aluminum addition, the $CeAl_2$ separation at the grain boundaries does not occur as thin connected layers, but as small individual crystals.

★*Aluminum*, Berlin, No. 4, 1943, p. 157 to 159.